VACUUM

Vol. 1 JULY, 1951 No. 3

VACUUM PROCESSING ECONOMICS

THE EXPERIENCED VACUUM WORKER now takes for granted the swift, certain and consistently produced high vacuum even in the most complicated large-scale apparatus. So dramatically have conditions changed from the time—only a few years ago—when vacuum, with limited exceptions, was confined to the Laboratory with tortuously assembled apparatus, almost non-existent pumping speeds, inaccurate instrumentation, leak hunting ever present and time consuming, that it is not surprising many potential vacuum users are still unaware or sceptical of the tremendous possibilities of industrial vacuum processing and that the semi-skilled worker has replaced the high vacuum research expert formerly required to run a vacuum system.

It is, of course, the purpose of 'Vacuum' to bring these facts constantly under review with reports on new applications and techniques, but we are not so mesmerised by the wonders of high vacuum that we do not appreciate the vital economic aspect. In many cases only the vacuum process can produce the required product and economic improvements must depend upon apparatus developments to increase output and reliability; there are, however, many vacuum processes now competing with older methods which can only succeed if economically attractive or yielding a superior product at some increased cost. Freeze-drying, for example, can dry even the most delicate materials which upon reconstitution are almost indistinguishable from the original, a result considerably ahead of other drying methods, but except for certain intrinsically costly products the process is not yet economically attractive.

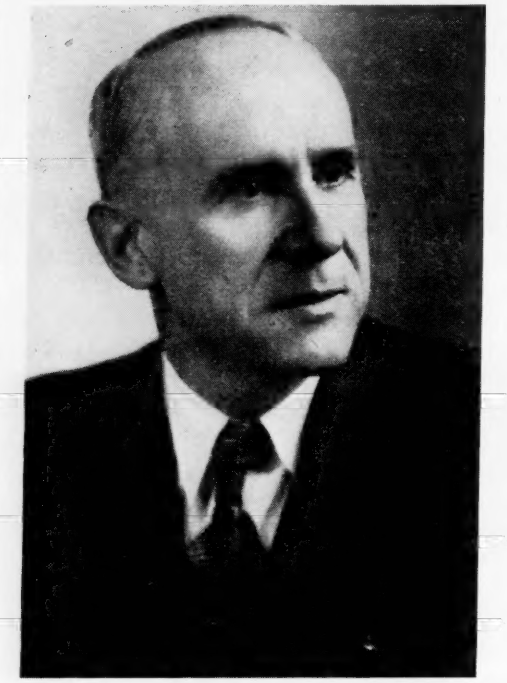
There is therefore a very real need for economic appreciation of vacuum processes, one result of which would be a stimulation in further developments to overcome any revealed economic deficiencies. As yet, few publications have appeared of this kind. We call one to mind, an article by M. Schneider in *Modern Plastics*, April 1950, where it was shown that apart from other advantages vacuum metallisation was also economically attractive. A further object of 'Vacuum' will therefore be to encourage the publication of economic surveys.

TITANIUM, ZIRCONIUM & VACUUM

THE PRODUCTION OF TITANIUM and zirconium on an industrial scale is a metallurgical development of far reaching importance. The properties of these materials, in particular those of titanium, are exceptional with regard to their tensile strength, resistance to corrosion and comparatively light weight. The uses of titanium in the fields of atomic energy, rearmament and aircraft construction—to quote only a few—are assured and spectacular figures are given as estimates of total world consumption. Plants are under construction in the U.S.A. which are scheduled to produce 3,500 tons of titanium in the year 1952 but it is said that this output will hardly cover the immediate requirements of government establishments in that country. Total annual production is expected to settle eventually at a rate between that of stainless steel and aluminium. Nothing authoritative is known on production plans regarding titanium in this country. We can only hope that the trend of economic affairs will not adversely affect the ability or initiative of our industry to play its part in these metallurgical developments.

The only process so far industrially established for the manufacture of these two newcomers to structural materials depends on vacuum technique, without which it would be improbable that we could talk today, for example, of 'titanium of a commercial grade of 99.5% purity'. Of course, vacuum methods have been used for a long time in metallurgy but considering the potential advantages the extent of their applications is still very limited.

The advent of titanium and zirconium has turned the spotlight on vacuum metallurgy. Vacuum engineering has proved once more—and this time conspicuously—its service to industrial progress. There are, it is true, many problems which require solving for the full exploitation of vacuum metallurgy and for this metallurgist and vacuum engineer should work together.



WILLIAM JUSTIN KROLL

Consulting Metallurgist

Corvallis, Oregon, U.S.A.

Dr. William Justin Kroll was born in Esch/Alzette, Luxembourg in 1888. He studied at the Technische Hochschule in Berlin-Charlottenburg and received his degree in 1918 after submitting a thesis on pure boron. After leaving the University he specialised in the study of rare metals. In the years 1918-1919 he developed methods for producing calcium lead by the reaction of sodium lead with calcium chloride and by reacting calcium carbide with lead; this latter process is the only method applied in practice today. During the same period he proposed the use of calcium as a scavenger for bismuth lead. The American Smelting and Refining Company developed this idea into an industrial process which is now the standard procedure. In 1924 Dr. Kroll founded his own laboratories in Luxembourg where he remained for another 16 years. In 1940 he went to the United States where he became associated with the Union Carbide Research Laboratories at Niagara Falls. In the year 1945 he undertook research work on behalf of the U.S. Bureau of Mines in Albany, Oregon.

Dr. Kroll's name is connected with a great number of outstanding achievements in the field of metallurgy, for instance, the fluoride reduction process for beryllium metal and the production of hot malleable manganese and chromium, but his main discoveries concern the production of malleable titanium and zirconium. The basic ideas underlying these processes were worked out in 1936, whilst he was still in Luxembourg, but their application on an industrial scale was initiated only after Dr. Kroll had joined the Bureau of Mines. The American titanium industry is expected to reach a capacity of 3,500 tons a year in 1952 and this figure will certainly grow to reach a few hundred thousand tons within our generation.

The nature of Dr. Kroll's work calls for the extensive use of vacuum, especially in connection with the separation of magnesium-chloride from titanium or zirconium sponge. Vacuum, therefore, as used in metallurgy is a subject with which he is exceptionally familiar.

Vol.
1
1951

VACUUM METALLURGY

its characteristics and its scope

W. J. Kroll *Consulting Metallurgist, Corvallis, Oregon, U.S.A.**

Summary

THE ACTUAL and potential uses of vacuum methods in metallurgy are discussed. The author makes a general examination of conditions in metallurgical processes carried out in vacuum. He then proceeds to survey existing furnace designs, their performance and limitations. Finally, he describes a large variety of metallurgical procedures, where the application of vacuum would appear to offer advantages.

Sommaire

LES METHODES ACTUELLEMENT employées en métallurgie et celles qu'il serait possible d'utiliser sont discutées. L'auteur examine d'une manière générale les conditions prévalant dans les procédés utilisant le vide. Il passe en revue ensuite les constructions de fours existants, leurs performances et défauts. Enfin il décrit une grande variété de procédés métallurgiques pour lesquels l'application du vide paraitrait offrir des avantages.

INTRODUCTION

IN METALLURGY, vacuum was used industrially for the first time during World War 1914-18. It was Messrs. Heraeus Vacuum Schmelze in Germany who did the pioneering work, inspired by the vision and ingenuity of Dr. W. Rohn.[77] The intention was to degas and purify metals and alloys, especially steels and nichrome, by melting *in vacuo*. Various types of furnaces were developed for the purpose. In the early designs capacities were of the order of 300 kg and the crucibles were resistance-heated by means of molybdenum windings. Later, three-phase low-frequency vacuum furnaces of the Roechling-Rodenhauser type were built with a capacity of up to 4 tons. Between the two World Wars, Rohn put a number of high-frequency vacuum furnaces into operation. The smaller types were built into a shell of resin-impregnated wood and the larger types—reaching a capacity of 5 tons—into a steel armature. The latter were provided with a separate high-frequency tapping coil and a deflection device controlling the direction of the jet during the tapping operation, in order to facilitate the casting of several ingots at the same time. However, the results were not worth the effort. The original aim of producing high grade chromel-alumel type thermocouple alloys of low gas content and consistent characteristics was achieved but at a price which made it questionable, whether the quality of the product was sufficiently superior to that obtained by established methods. Beryllium copper was degassed for some time in small high-frequency vacuum furnaces, but this process was soon abandoned, as it was found that gas-free metal could be obtained by simpler means. Ferro-chromium and stainless steel were decarburised with oxides such as Cr_2O_3 and especially, with magnesia-bearing slags. Here again, operations were discontinued. To quote a final example, it was said for a while that the degassing of large batches of steel has the advantage of producing castings with an elongation and impact strength sufficiently high to eliminate hot working, but no concrete results have ever been made known.

This introduction to the history of vacuum melting may serve as a lesson to all those who think that vacuum methods can be applied as a 'cure-all', whenever difficulties arise in the processing of metals. Obviously, the question of the proper uses and real

*M.S. received July, 1951.

benefits of vacuum in metallurgy deserves close scrutiny.

Pyrometallurgy offers many opportunities for the application of vacuum but only in a few instances have vacuum methods been finally established. Firstly, in a great number of processes it is required to protect the metal against oxidation and nitriding by air or to provide for the elimination of gases, e.g. hydrogen, which may be present in the metal. Mention should be made here of degassing with the help of carbon which reacts with oxides more readily at reduced than at atmospheric pressure. Secondly, there is the wide field of metal purification and separation of alloys by volatilising obnoxious alloy elements. This field has barely been entered despite the fact that it contains many possibilities. Thirdly, one could possibly apply vacuum in separating inorganic compounds for the purpose of effecting the dissociation of the compounds or generally a type of reaction which yields a condensate or residue containing useful dissociation or reaction products. Finally, metal winning by the Guntz method should be quoted. This process consists of reducing the oxide of a volatile metal in a vacuum with a less volatile reducing agent, recovering the evaporated metal by condensation. On the other hand, reduction processes are known which provide for the compound formed to be volatilised.

In the course of this study it will be necessary to examine some of the basic features of vacuum methods, in particular, those which apply to the treatment of metals at high temperatures. Also, the factors governing the design of furnaces required for a variety of specific purposes (e.g. the materials from which they are built) and the heating systems employed will have to be considered since the use of vacuum imposes restrictions not ordinarily encountered in operations at atmospheric pressure.

PHENOMENA OBSERVED IN VACUUM FUSION[1,44,83]

The hydrostatic pressure within a metal bath has an important bearing on the evaporation, as can be seen from the following considerations.

Assuming a mercury bath is kept at the room temperature of, say, 19.6°C—a figure chosen to facilitate the easy conversion of pressure factors—then, its vapour pressure is 1.14 micron. According to the temperature-vapour pressure table of mercury the respective value for 7.1°C is 0.36 micron. In the temperature range from 7.1 to 19.6°C, therefore, the vapour pressure increases at a rate of 0.0624 micron per 1°C. If evaporation is to take place in the bath at a level 10 mm. below the surface (=10 mm. Hg hydrostatic pressure) the temperature at this level would have to be 188°C. The bubbles formed at this level would expand about 7,000 times before they reached the surface of the bath. Boiling or bubble formation is the result of uneven heat distribution and, since metals are good heat conductors, bubble formation will not take place in a bath which is kept under vacuum, unless heat is supplied to it at an unreasonably high rate.[44] In other words, in vacuum, evaporation takes place at the surface of the bath only and, therefore, it is necessary to increase its surface, where rapid evaporation is required for a given heat input. This can be achieved by dropping the metal in shower form through the evacuated space or by the provision of metal cascades or similar arrangements.

However, a temperature gradient is necessary as is the case in all operations involving a heat transfer. Thorough mixing of the bath is indispensable as the metal at the surface transmits its heat very rapidly to the vapour. There are instances, where evaporation has to be carried out at a sharply defined temperature level, for example, in the fractionation of certain alloys. Here, good stirring is of particular importance because of the local depletion of the alloy at the bath surface which has a direct effect on evaporation conditions.

In passing we will now compare these conditions with those prevailing in evaporation under high pressure. Over the temperature range of 450-500°C. the vapour pressure of mercury rises from 4.25 to 8 atmospheres, i.e. 57 mm. per 1°C, and at a point 10 mm. below the surface of the bath the temperature is 0.175°C higher than at the surface itself because of the hydrostatic pressure. Such slight temperature

Vol. 1 1951

differences may well exist, even in a metal bath which is effectively stirred. Consequently one can expect the bath to boil. This is still more pronounced with liquids of low specific gravity and low thermal conductivity such as water and, in fact, observations made with high pressure 'Benson' steam boilers have confirmed that boiling takes place under these conditions in the form of very fine bubbles dispersed throughout the bulk of the hot water.

Naturally, all other types of gas evolution which are a function of pressure and temperature depend at the same time on the hydrostatic pressure or, in other words, on the level of the bath, at which the evolution takes place. This applies, especially, to reactions between carbon and oxides, dissociations of compounds such as hydrides and the liberation of dissolved gases.

At a given temperature, reactions between carbon and oxides, especially those of the highly exothermal type can be carried much further in vacuum than at atmospheric pressure but much depends on the manner, in which the oxide is offered to the carbon. Provided the bath is well stirred, the dissolved oxide will react readily but at the surface of the bath only. The oxide in the slag or in the furnace lining below the surface of the bath might not react at all because of the hydrostatic pressure. As the gas evolution is confined to the surface, intensive stirring is essential in all purification processes carried out in vacuum. This requirement was recognised very early. Today, low- and high-frequency furnaces are usually fitted with mechanical mixers or similar arrangements to keep the bath turbulent.[2,3,77]

Whether it is possible to carry out such purification processes at very low pressures, i.e. in the micron range, depends very largely on the pressure exerted by the metal vapour itself at the temperature required. Iron may serve as an example : Its vapour pressure at the melting point is about 50 micron. At this temperature, the pressure of carbon monoxide over certain highly exothermic oxides such as beryllia might be the same, and the dissociation pressures of these oxides might be of the same order, too. Where these conditions apply, the partial pressure of the evaporating metal can be expected to cause considerable interference with the actual process of purification. On the other hand, the use of a very high vacuum has recently produced remarkable results in the deoxidation and degassing of iron-chromium alloy containing 28% chromium. To counteract brittleness the material was melted in a high vacuum and showed good impact strength after treatment.[4,5,6]

So far, discussing the conditions in the hot crucible, from which the metal evaporates, the term 'pressure' has been used somewhat indiscriminately. Actually, there are two pressure components resulting from the presence of two types of gases, i.e. condensable and non-condensable gases. For the purpose of this study water vapour will be included in the latter. The former type represents mostly vapours of metals or compounds which, on cooling, condense inside the furnace and therefore, never reach the pumps, whereas non-condensable gases are carried on to the pumps. Non-condensable gases may be released from the metal (e.g., H_2,N_2,CO,CO_2 and SO_2) or liberated from structural parts of the furnace (e.g., H_2O and CO_2). Or, they may have 'leaked' into the furnace and thus contribute to the total pressure prevailing in the crucible, unless they react with the metal. The gauges usually attached to the furnace walls measure, of course, only the pressure of the non-condensable gases and in some cases, depending on the calibration or type of gauge, they may fail to indicate the pressure of the water vapour. In our case, where we wish to establish the pressure of condensable metal vapours, this method of measurement is as little effective as that of determining the temperature inside a room by hanging a thermometer outside the window. However, the error incurred by measuring only part of the total pressure in the chamber, i.e. the partial pressure exerted by the non-condensable gases, might not be very great if the latter is high compared with that of the vaporised metal. For instance, iron is usually melted at a residual gas pressure of about 5 mm. Hg measured in the manner described, whilst its vapour pressure at the melting point is about 50 micron or 1% of the total. Where metals have a high vapour pressure at their melting point—for example, magnesium

=2 mm. Hg and calcium =1.8 mm.Hg—measurement of the (usually) low pressure prevailing in the space surrounding the crucible gives no proper indication of evaporation conditions but it provides information on plant conditions (re. leaks, etc.) and on the amount of non-condensable gases evolved from the metal.

CONDENSING THE VAPOUR

The transfer of the vapour from the surface of the bath to the condenser results from a pressure difference, the magnitude of which governs the evaporation rate. Figure No. 1 shows a vacuum retort as used by Pidgeon in the silicon-dolomite process.[7] The vapours in the retort consist of superheated magnesium rising at a pressure of about 18 mm. Hg from the reaction mixture of 2(MgO.CaO) + Si heated to a temperature of about 1,150°C. This pressure is far in excess of that normally required for the successful evaporation of metals, but necessary in this case for reasons discussed below. In ordinary circumstances a vapour pressure of 1.8 mm. Hg is sufficient to cause the transfer of the vapour at a rate, rapid enough to ensure that production on an industrial scale is a commercial proposition. This statement is based on the fact that calcium which can be distilled as well as sublimed, has at its triple point a vapour pressure of 1.8 mm. Hg and at this pressure the vapour is transferred at an industrially acceptable rate as long as a vertical retort is used with no obstructions in the path of the vapour. One could, of course, operate at lower vapour pressures (see below, calcium reduction) but the process would take longer and might not pay, except in the case of plant designed for the evaporation of metals on a small scale (e.g. coating plant). The value of 1.8 mm. Hg represents the minimum pressure required. In all cases, where obstructions are placed in the path of the vapour, higher pressures are needed. In the Pidgeon retort, for example, the vapour rising from the briquette has to overcome first the resistance offered by the briquette itself and the batch of material in general. Secondly, it has to pass baffles and heat screens mounted in the retort.[49] In addition, a certain amount of non-condensable gases such as hydrogen are liberated during the process. This explains why, in this case, a pressure as high as 18 mm. Hg is employed in production.

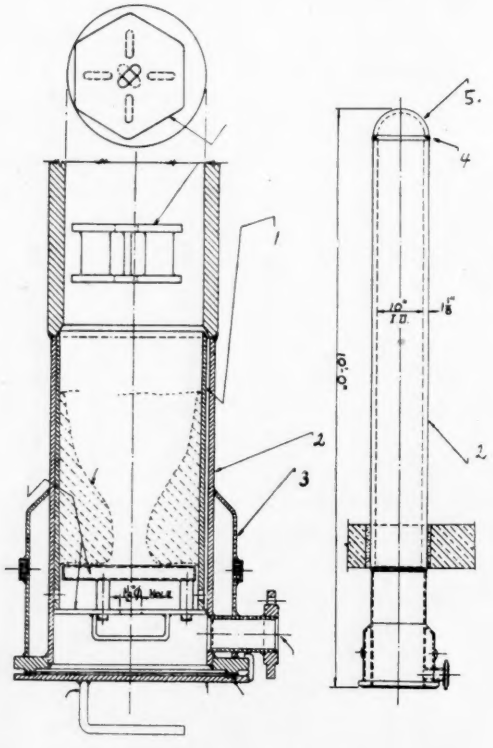


Fig. 1. Pidgeon Retort[7]

Explanation of numbers :

1. Condenser shield.
2. Retort.
3. Water jacket.
4. Weld.
5. Bottom.

The temperature and pressure conditions along the vapour path from bath surface to condenser will now be considered. Figure No. 2 shows that the metal vapour is in a saturated condition when it leaves the evaporating surface but passing the hot walls of the crucible it will take up more heat and become superheated. Thus, the vapour can travel a considerable distance before condensation occurs. As its temperature falls, it becomes saturated again and finally constitutes a wet vapour depositing as a

Vol. 1 1951

condensate on the cool surfaces. The drawing shows the probable pressure distribution in the vapour column over the whole distance from evaporating surface to condenser. In certain conditions not yet fully explored, no wet vapour will form. Instead, solid metal crystals are precipitated on to the cool surfaces like snow. If this phenomenon could be brought under control, there would be a chance of producing metal powders directly from the vapour phase.

The physical state of the condensate is a function of the pressure conditions in the condenser. Metals which have a high vapour pressure at their melting (triple) point can be condensed in liquid form, if due care is taken that temperature and pressure inside the condenser are appropriate. In the case of magnesium, for example, one would have to operate in a region above 650°C—the melting temperature of the metal—and at a vapour pressure exceeding 2 mm. Hg to facilitate condensation in liquid form. As yet no use has been made of this possibility in the magnesium-dolomite process, despite the fact that a pressure as high as 18 mm. Hg is available in this case. It has been proposed, however, to condense barium in liquid form and to collect it through taps. This latter suggestion has been put into practice.[51,86]

Solid condensates grow in a direction opposite to that of the heat flow. If the condenser surface is hot, the deposition takes place in the shape of large crystals of a columnar structure or trees of great density as a result of the thermal mobility of the condensing particles. If the condenser is cold, a powdery deposit is usually obtained. Another factor influencing the structure of the condensate is the actual processing time. At the start of a run, when the pressure of the residual (and frequently chemically active) gases, such as CO_2 and H_2O, is high compared with that of the metal vapours present, a thin powdery layer is deposited in the first instance on the walls of the condenser. Later, when evaporation takes place at the full rate, the non-condensable gases have only a minor part to play and, as a result of the great volume of metal vapours present large dendrites develop. In the final phase, when the conditions revert to those characterising the initial

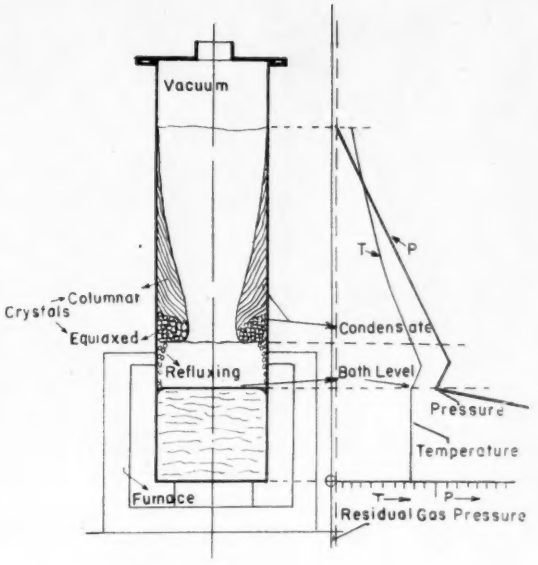


Fig. 2. Distillation of metals in a vertical retort. Diagram of temperature and pressure along the axis.

stage of the run, another powdery layer will be deposited on top of the dendrites. If the vacuum is poor due to leaks or other deficiences in the plant, the metal vapours will oxidise during condensation or nitrides will form and result in a pyrophoric ' blue powder ' type of product.

Condensation in solid form can be effected in an upward direction and in solid or liquid form in a downward direction. The deciding factor is the vapour pressure of the metal at its melting point. Liquid condensation in an upward direction causes refluxing, in particular, in the hot area between bath and condenser. Table No. 1 shows the vapour pressures of various metals at their melting point and the temperature ranges, where refluxing takes place, if the process is carried out at the ' industrial ' vapour pressure of 1.8 mm. Hg. It will be seen that refluxing on a substantial scale can be expected in the case of almost all elements given in that table other than Mg, Ca, Cr, Sr, As and Mn.

Sometimes one has the choice of providing for condensation in liquid or solid form and it is, therefore, worth while to establish the advantages and

disadvantages of both these methods. In the case of condensation in solid form, condensation in an upward direction is usually arranged for, on the assumption that the free passage of the metal vapour is not obstructed by the condensate at any time. In the event, however, the area available for the passage of the vapour narrows down, pressure is built up and the bath underneath overheats until lower parts of the condensate melt and drop into the superheated bath. As a result, a sudden rush of overheated metal vapour will surge into the condenser and cause further melting of the condensate. Similar conditions are obtained, if the pressure built-up is originated by the dissociation of an inorganic compound such as a hydride which causes the sudden evolution of a great quantity of gas. This is a very frequent occurrence in the distillation of barium for purification purposes.

Another problem connected with condensation in solid form is that of heat conduction within the condenser. The heat developed in the condenser dissipates through the steadily growing condensate to the cooled outside walls. If the heat supplied to the metal bath is constant throughout, evaporation will be affected by the thermal conduction properties of the condensate which change with the thickness of the deposit. The situation is aggravated by the fact that the heat tends to concentrate on the steadily diminishing surface of the condenser itself. In these circumstances it is extremely difficult to effect good fractionation. There is the additional danger that a heat gradiant might develop along the axis of the condenser, if the latter is not held artificially at a constant temperature over the whole of its length. It is, indeed, difficult to imagine that a condenser placed in the vicinity of a bath of fused metal could be held at a uniform temperature. For these reasons it is obviously no easy task to arrange for accurate fractionation in the case of condensation in solid form. However, where metals have widely differing vapour pressures, as for instance in the case of sodium and magnesium, good separation has been achieved.[52]

Liquid condensation is less difficult. It offers the major advantage of accurate temperature control due to the fact that the free passage of the metal vapours is maintained and an even heat dissipation through the thin film of condensate is assured. It is usually carried out in a downward direction either in a vertical or in an inclined retort. If desired, arrangements can be made for the metal to be frozen in the bottom part of the apparatus underneath the actual condenser.

Vol. 1 1951

Table I **Pressure and Temperature Data for Metal Evaporation**

Metal	Vapour Pressure at the Melting Point mm. Hg	Melting Point °C	Temperature at the Minimum Commercial Pressure of 1.8 mm. Hg	Refluxing Temperature Range in °C at 1.8 mm. Hg	Metal	Vapour Pressure at the Melting Point mm. Hg	Melting Point °C	Temperature at the Minimum Commercial Pressure of 1.8 mm. Hg	Refluxing Temperature Range in °C at 1.8 mm. Hg
Mg	2.5	650	620*	none	K	nil	63.6	364*	300.4
Ca	1.8	850	850*	none	Sn	nil	232	1400	1168
Cr	60.0	1920	1350	none	Mn	1.0	1244	1300	56
Fe	0.05	1535	1810	275	Al	nil	659.8	1520	860.2
Ni	0.01	1455	1930	475	Be	0.1	1300	1590	290
Ag	0.002	960.05	1340	379.5	Si	0.02	1450	1680	230
Zn	0.1	419.47	505*	85.53	Pt	nil	1773.5	2660	886.5
Pb	nil	327.4	1020*	692.6	Mo	0.04	2600	3020	420
C	170 atm	3727	3120	none	W	0.04	3380		
Cu	0.00001	1084	1630	546	Bi		271.0	960*	689
Li	nil	180	780*	600	As		818	480*	none
Ba	0.08	710	850*	140	Sb	0.01	630	1100*	470
Sr	1.0	770	780*	10	Hg	nil	−38.87	138*	176.87
Na	nil	97.8	450*	352.2	Se	0.009	217	400*	183

All data approximate only.
Nil = 0.000001 or less
* = Metals that can be distilled in present day industrial equipment made of iron or heat-resisting alloys.
Data taken from Van Arkel, *Reine Metalle*, and Espe-Knoll, *Werkstoffkunde de Hochvakuum Technik*, (Springer Verlag, Berlin).

All the tools now employed by the oil industry in fractionating distillation could be used for the treatment of metals as well i.e., column stills, falling film stills and the like. But so far, no attempt has been made in that direction. The effective separation of mixed metal vapours is still handicapped by the lack of suitable equipment.

EVAPORATION FROM THE SOLID OR LIQUID STATE ?

Some attention should be given to sublimation at this stage. Evaporation from the solid state has certain advantages over evaporation from a liquid bath. For example, by subliming solid turnings or comminuted metal the evaporating surface can be greatly increased and, therefore, comparatively low temperatures could be employed without endangering the precision of the separation. On the other hand, it is difficult to obtain a good heat transfer in a solid batch of material due to the low temperature gradient. As the more volatile components of the processed material leave the surface, replacement is effected by diffusion from the core. This is a slow process, especially at low temperatures. Finally, there is the danger that non-volatile impurities may form a crust and interfere with the evaporation of the volatile component. Of course, where contamination is low, such interference might be negligible. Magnesium of the commercial grade for instance, can be purified by sublimation without difficulty.

The temperature and pressures employed in a vacuum process very largely depend on the purpose of the operation. Melting, for example, usually accompanied by a purification effect, can be performed in an open crucible only if the vapour pressure of the metal is low at its melting point. Iron and copper can be melted in an open crucible but the melting of metals with a high vapour pressure requires the provision of a comparatively high pressure in the chamber to counteract losses by evaporation. If evaporation takes place the metal might cool down to the freezing point, unless the heat input is great enough to make up for the losses. This can be overcome by covering the crucible tightly, thereby creating the required metal vapour or gas pressure inside. Alternatively, one can introduce a noble or inert gas into the vacuum chamber by means of a leak-in device. Time is an essential factor in melting. If arrangements have been made for the heat input to be sufficiently rapid, one can melt all metals in an open crucible or sleeve even those with a relatively high vapour pressure at their melting point. This has been proved in practice by melting tantalum in an arc under vacuum.

PLANT MATERIALS, HEATING SYSTEMS AND FURNACES

Retorts used at atmospheric pressure can be built from iron only as long as the operational temperature does not exceed 500°C. Otherwise excessive scaling takes place. But iron can be used for temperatures up to 700°C, if it is sprayed with a coat of stainless steel or aluminium. At higher temperatures, i.e. in the region of 700 to 1,200°C, iron has to be protected with a sheath of thin inconel or a coat of glass.[9] Placing the hot iron shell into an evacuated furnace chamber (Figure 3) eliminates the danger of oxidation and avoids any action due to atmospheric pressure.[10]

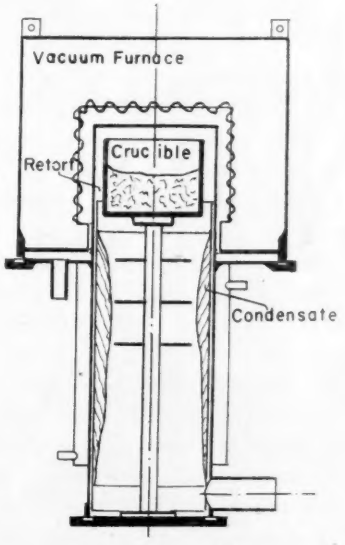


Fig. 3. Salt separation vacuum furnace used in producing titanium and zirconium sponge.[10]

At about 800°C mild steel shows creeping to a considerable degree and it is preferable to use stainless steel in this temperature region. However, in the range of 1,100—1,200°C creeping is equally pronounced with both materials. Another matter which requires serious consideration is the fact that iron is liable to be attacked chemically. This danger is very closely connected with the thermal stability of inter-metallic iron compounds in vacuum. So far very little is known on this subject. Iron does not seem to form a compound with Zinc in vacuum at temperatures above 500°C, but its S-, Se-, Ta-, As- and Sb-compounds are probably stable at all temperatures up to 1,200°C. If this was confirmed, iron could no longer be considered for the construction of vacuum furnaces designed to operate at higher temperatures in the presence of these elements or their compounds. Heat resisting Nichrome-nickel alloys are known to react in vacuum with calcium and magnesium vapours at 1,200°C. The facts are that Nichrome heat-resisting steels and iron are the only materials which are now used for the construction of large industrial vacuum plant. Thus, the operational temperatures of industrial vacuum processes are restricted to a maximum of 1,200°C. Column 4 of Table No. 1 gives all metals which could successfully be evaporated at a vapour pressure of 1.8 mm. Hg in the permissible range of temperatures. Danger from chemical action is not considered in this connection. It will be noted that apart from the alkali and alkaline earth metals, lead is very suitable for purification under vacuum in such equipment.

Vacuum plant materials for use at temperatures beyond 1,200°C are scarce. Graphite can be employed as a crucible or heating element, but its application is limited, because it reacts with almost all refractories and oxides above 1,300°C[11] and the CO/CO_2 formed as a result of these reactions has an adverse effect on the process. Graphite is actually used for resistor elements in furnaces operating above 1,200°C, for instance, in calcium reduction furnaces[53] and it would appear, therefore, that carbon is stable in calcium vapours at high temperatures and sub-atmospheric pressures. Silicon and aluminium[88], in similar conditions, attack graphite and form carbides, but it is said that silicon carbide resistors can only be used up to 1,400°C in vacuum.[15] This seems to prove that the thermal stability of carbides in a vacuum needs further investigation. At temperatures above 1,600°C and at very low pressures carbon evaporates and this can be the cause of considerable difficulties. The migrating carbon vapours might react with hot refractories and cause the formation of carbon monoxide. Where siliceous refractories are used, the volatile monoxide SiO might also form (see Table No. 5). Both these gases would probably pass over the metal bath. If the latter contains a very active metal such as titanium or zirconium, it will absorb silicon, carbon and oxygen from these gases. To prevent this, the refractories should be protected with molybdenum sheeting and the graphite enclosed by a container of the same metal.[13]

Tungsten, molybdenum and tantalum can also be used for resistor elements in vacuum, but are still more troublesome than carbon. Tungsten and molybdenum tend to become brittle at high temperatures. All three metals are sensitive to oxygen, and tantalum, in addition, to nitrogen and hydrogen. Another factor limiting the use of these three metals is cost.

Finally, conducting refractory oxides, such as ZrO_2, have been employed in high frequency vacuum furnaces of laboratory size.[54]

The design of furnaces very largely depends on the temperatures to be obtained and on the heating systems to be used. Retorts[7] are normally heated externally, but the charge can also be heated electrically from inside.[17] In the case of arc melting the material is placed in a water-cooled copper sleeve and the arc struck against the metal.[16,18] Where it is necessary to produce very high temperatures, high-frequency furnaces offer the greatest advantages. If the charge is non-conducting, conducting sleeves have to be used.

Wherever vacuum furnaces are heated electrically, one has to deal with the peculiar phenomena of electrical discharges in an evacuated vessel.[1] A plain tungsten tip, for instance, in a good vacuum starts to emit electrons at about 2,300°C. If the tungsten tip is coated with thorium this temperature is reduced to 1,427°C and in the event of oxide-

Vol. 1 1951

coated electrodes being used, emission starts at 500°C.[19] The electrons tend to ionise the gases in the evacuated chamber and thus render them electrically conductive. As a result sparking might occur at a voltage as low as 83 (the figure for barium-coated electrodes in a xenon atmosphere[20]). The sparking potential is different for each gas and varies with the prevailing pressure.[21] Fortunately, helium and hydrogen which are frequently used to provide an inert atmosphere, have high sparking potentials but in the case of argon great care must be taken, if it is admitted to the chamber at a pressure near the critical value.

Arc furnaces operated at sub-atmospheric pressures are particularly delicate to handle in this respect. If the residual gas in the evacuated chamber consists mainly of argon at a pressure near the critical value, a glow discharge will develop and spread through the chamber. Calcium or lithium vapours rising from the bath are known to cause a glow discharge which usually leads to the rapid destruction of the electrode support at the point where the discharge originates. Whenever argon is being leaked into the vacuum chamber this condition should be borne in mind and the pressure of non-condensable gases in the chamber should be kept sufficiently high to avoid the formation of a gas discharge at a voltage close to the arcing potential.

One might think that heating by means of an arc represents the most efficient method, for the heat is generated precisely in the area where it is most needed, i.e. at the surface of the bath. In fact, the arc heats only a very restricted space and if too much energy is conveyed to the area, those constituents of the bath which it is not desired to evaporate may be caused to volatilise.

Resistance and high-frequency furnaces work reliably in air at 220 volts. Atmospheres of hydrogen or helium may be employed equally well but difficulties have been experienced, where the heater element was placed on the inside of the vacuum chamber. To quote an example, it has been found that magnesium chloride vapours rising from the bath in such furnaces became ionised.

High-frequency furnaces of the type consisting of a quartz tube carrying the energising coil on the outside and operated at a high voltage are often unsatisfactory in the event of the pressure in the tube reaching the critical value for the gas present. Heating is then limited to the ionised atmosphere and the bath itself cools down.

Melting in a vacuum arc is successful if the arc is confined to the vapour column between the electrodes formed by the evaporating metal. A low voltage arc will not ordinarily spread through the furnace chamber because the fringes of the vapour column cool down due to expansion and other effects which combine to make the fringes non-conductive. On the other hand, a high voltage arc might heat the gases surrounding the electrode so intensely that their pressure approaches the critical value. The arc would then be transformed into a glow discharge spreading through the furnace chamber and heating the whole space. It has been proposed to utilise this effect in the distillation of metals[22] by providing for the production of heat in the metal vapour column above the bath. It is doubtful whether such a glow discharge furnace would be economical in operation, as most of the heat is generated in the region of the vaporised metal and not in the fused metal, where the heat is most essential.

ESTABLISHED AND POTENTIAL USES OF VACUUM METALLURGY.

The Melting of Metals

Zirconium and titanium sponge are frequently melted and cast in a vacuum.[10] This process can be carried out in a split-tube graphite resistor-high-frequency, or arc vacuum furnace and results in the removal of hydrogen. Magnesium and magnesium chloride are removed at the same time, if they are present in the sponge. In connection with the manufacture of tungsten targets for X-ray tubes, copper is melted in vacuum and embedded in graphite by a casting procedure. Melting and casting of steels in vacuum might become an important feature in the production of low-watt carbon-free silicon transformer steel or in the decarburisation of stainless steel. Vacuum methods effect the simul-

taneous removal of hydrogen, phosphorus, sulphur and manganese content down to a very low percentage, but where manganese is concerned this might not be desirable. The melting in vacuum of steels containing 28% chromium results in an increased impact strength. This is attributed to the elimination of hydrogen in the process, although the part played by hydrogen in chromium steels has not been fully explored.[4,5,6] The decarburisation of ferro-chromium in vacuum was suggested many years ago, when it was realised that reactions of carbon with oxides which it is difficult to reduce by other means could be carried much further in vacuum than at atmospheric pressure, at a given temperature. The alloy can be decarburised in powdered form by adding magnesia[58] or silica.[55,56,57] Alternatively, its own oxide can be added[4,5,6] or produced by oxidation on the surface of the bath.[55,56,57] Vacuum melting with chromium oxide and magnesia slags achieves similar results.[77] Cast iron thoroughly deoxidised and desulphurised by vacuum melting shows nodular graphite after freezing without the addition of dopes.

The Separation of Metals

Tables No. 5 and No. 6 show that many inorganic compounds will not dissociate in vacuum. Instead, they have a well-defined boiling point of their own. Does this condition apply to intermetallic compounds to the same extent ? Asking this question may lead to an attempt to make a distinction between the two types of compounds but it would prove very difficult, indeed, to think of a correct definition for the latter. However, it can be said that the majority of intermetallic compounds is rather unstable. Their presence in an alloy is not usually indicated, as one might expect, by the temperature composition curve for a given pressure. In the case of binary systems containing stable compounds one might think[42] that the temperatures at each end of the diagram would rise until they reached a maximum, which for a given pressure, corresponded to the temperature of the intermetallic compound, but no such cases have yet been reported. Compounds are known to exist in fused alloys, e.g. the compound Na_3Hg boils off without decomposition.[80] Where the chemical bonds are very strong, irregularities appear in the activity isotherms and considerable deviations from the Raoult law have been observed, but of course deviations of a similar order, positive or negative, have been found in the case of many alloys which form no compounds at all : see Table No. 2.

Our knowledge of the chemical structure of liquid alloys is still only elementary[59-64] and much more information will have to be gathered first, especially on alloys containing highly exothermic compounds, such as AlNi and Bi_2Ca_3, before equilibrium and activity diagrams can be properly correlated.

Figure No. 4 shows the relationship of pressures and temperatures in the binary aluminium—zinc system which contains the compound Al_2Zn_3 (Zn=78.4%). Here, an actual attempt has been made to correlate equilibrium and activity diagrams.[59-64]

Processing on the industrial scale is usually carried out at low pressures of the order of 1.8 mm. Hg or more and it would be an advantage to know the corresponding temperatures for the whole composition range of each system. Unfortunately, information in this respect is lacking. Most investigations published give temperature values which

Table II **Deviations from the Raoult Law[46]**

Degree of Deviation	SnCd	AgAu	PbZn	BiPb	BiSn	BiTl	MgPb	CdPb	AlZn	CuCd	CuZn	AgZn	AgCd	AuZn	AuCd	AlMg
Large	●		●			●	●	●	●	●	●	●	●	●	●	
Medium		●		●												●
None					●											
Compound Present	no	no	no	no	no	yes	yes	no	yes	yes	yes	yes	yes	yes	yes	yes

Vol. 1 1951

apply to much higher pressures.

Turning to ternary and multi-component alloys we find that any one of the addition elements can produce either improved stabilisation or increased volatilisation. For example carbon stabilises manganese in iron alloys in the form of carbide and the evaporation of manganese from a carbon-free ferro-manganese alloy proves less difficult than from the same alloy containing carbon. On the other hand, silicon mono-sulphide contained in cast iron is vaporised before iron and iron sulphide and can thus be used as a desulphurising agent. Stable compounds frequently found in distillation residues are oxides, nitrides, silicides and carbides such as Al_2O_3, SiO_2, CbN, TiC, $CaSi_2$, FeFi but the degree of dissociation achieved depends on the temperature, at which the process is carried out and, as the temperatures of the various distillation procedures differ, certain compounds are retained in some of the alloys and not in others.

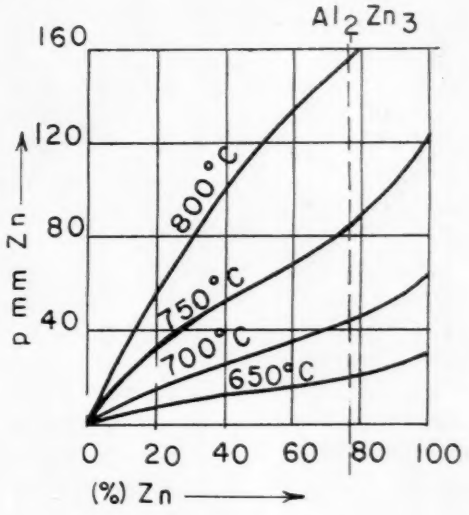


Fig 4. Pressure temperature relations in the system Mg/Zn according to Schneider.[59-64]

On heating a mixture of immiscible (layer forming) metals in vacuum, the layer of the heavier metal is subjected to the hydrostatic pressure of the layer formed from the lighter metal. Where the values of the vapour pressures of the metals present in the mixture are close to each other, the heavier metal may not be vaporised at all, until the upper layer has completely evaporated, although its vapour pressure is higher. Naturally, if the vapour pressures of the metals involved are sufficiently far apart, the vapour of the heavier metal will bubble through the layer of the lighter metal.

It should be noted that the dissociation of compounds complicates the condensation process. Copper oxide dissociates at the 'industrial' pressure and temperature required for rapid volatilisation, i.e. at 1,600°C and 1.8 mm. Hg, but the melting point of copper is only 1,084°C. Thus, the condenser is appreciably cooler than the bath and this condition results in partial recombination of the dissociated oxide. Due to the rapid rate of cooling, however, a certain amount of oxygen does not recombine and is carried on to the pumps as can be demonstrated with a glow discharge tube. Recombination phenomena similar to the one just described can be observed frequently. They can prove very troublesome, for instance, in connection with the determination of oxygen in steels containing a large amount of manganese. Manganese vapours reaching the condenser may react with carbon monoxide originating from the bath. Similar phenomena occur in the treatment of alloys with a high silicon and aluminium content, if heated in an oxide refractory vessel. Lower oxides[43] are evolved which, after condensation, tend to react with oxygen compounds in the vapour state.

In view of the fact that alloy constituents can contribute either to the stability of the compound or its volatility, it is impossible to predict the conditions obtaining in processes concerned with the separation of ternary or multi-component alloys. Table No. 3 gives a summary of the results of work carried out on this aspect of alloy treatment but refers mainly to alloys of the binary type.

In the purification of lead, separation is good with regard to Zn, Sn, Ag, As and Cu, fair with regard to Sb, and poor with regard to Bi. Sn, Sb, Ag and Cu are retained in the residue, Zn is part of the distillate.

In the case of tin purification, separation is good with regard to Pb, As and fair with regard to Fe, Sb, Ag. Fe and Cu remain in the residue, whereas Pb, As, Sb, Ag and As distill off.

Finally, copper purification shows poor separation of Sb, Sn, sulphur and oxygen. Antimony tends to distill off.

Vacuum methods for the separation of alloys were applied in Germany during the last war, especially in connection with the reclaiming of aluminium, magnesium and zinc from scrap. These processes consisted essentially of mixing the aluminium scrap with zinc (Zincal or Foundaminski-Loewenstein process[65,66,67]) or magnesium (Beck process[68]) followed by filtering through a refractory bed at a controlled temperature. The volatile solvent, zinc or magnesium, was recovered by distillation in vacuum furnaces of the carbon resistor- (see Figure No. 5), low-frequency- (Figure No. 6) or arc type. The Zincal process proved difficult due to the high temperatures required for the thorough removal of the zinc from the aluminium. The magnesium process was carried out with some measure of success.

The separation of zinc contained in brass under vacuum has been attempted several times, first during the world war 1914-1918 and again during the last war. It would appear, however, that it is more convenient to operate at atmospheric pressure on the lines suggested by Poland[69] which allow for easy charging and tapping.

A vacuum method for the de-zincing of lead after de-silverisation was recommended many years ago[76], but has only recently been put into practice. It seems to be firmly established now.[2,3]

The de-zincing of crusts forming in the de-silverisation procedure is in the experimental stage.[45,76] The experiments offer very good prospects. The crusts do not oxidise and therefore the loss of precious metal is very low, indeed. The distillation residue, depending on its composition might possibly be cast as anodes for direct electrolysis, permitting in this way a very rapid turnover of noble metals.

The distillation of manganese in vacuum yields a very pure material suitable for use in the manufacture of ductile manganese alloys but this method cannot compete with the electrolytic process.

It has been known now for quite some time[76] that lead can be eliminated *in vacuo* from tin and its alloys but no practical attempt has ever been made in that direction. It should be easy to construct a refining furnace which is fed and tapped continuously by means of barometric columns.[47] As iron reacts with the processed material, the hot parts of the furnace in contact with the tin alloy would have to be made from graphite. Where it is intended merely to change the tin concentration of the alloys to adjust their lead content in accordance with commercial requirements, operational temperatures could be restricted to the region below 1,000°C. It is certain that a process of this nature would fill a gap now existing in the production techniques employed by the solder and babbit metal industry. No doubt, the de-leading of high-duty tin-copper-antimony babbit containing alloys[48] is of considerable interest. Unfortunately, it has been found that vacuum methods could not be applied to the purification of Bolivian tin (Pb, Bi) due to the fact that it contains a number of impurities of comparatively low volatility.

Zinc can be purified under vacuum at temperatures close to its melting point. At the pressures and temperatures required for the process iron is unfortunately attacked by zinc. Otherwise it would have been advantageous to employ iron for the construction of the equipment.

It was found impossible to remove calcium from de-bismuthising dross. The same applies to the removal of lead from magnesium produced by the method involving the quenching of this metal in gases coming from an arc furnace, in which magnesia is reduced with carbon. Attempts to remove small amounts of sodium from lead by a vacuum distillation procedure have been equally unsuccessful.[74] The separation of calcium and lead by similar methods was found not to be practical either, the reason being that the intermetallic compounds which had formed in these cases dissociated in part during the course of the distillation and formed mixed vapours.

It has been suggested to de-bismuthise copper in

Vol. 1 1951

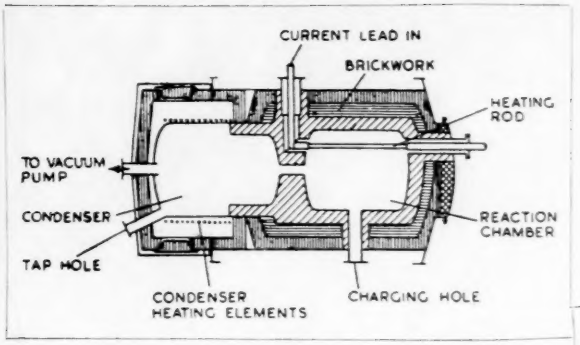


Fig. 5. Graphite rod resistor vacuum furnace of the I. G. Farbenindustrie.[88] Top View.

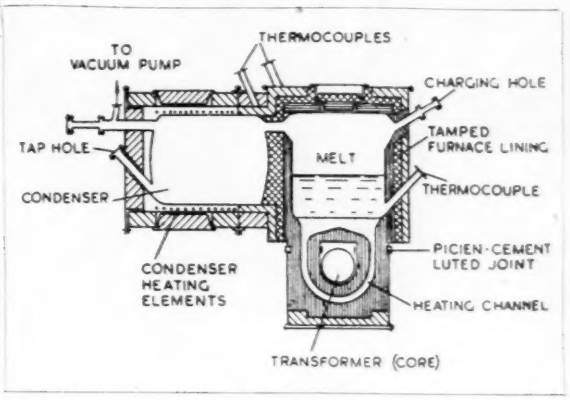


Fig. 6. Low-frequency vacuum furnace used by the I. G. Farbenindustrie for distilling metals.[88] Side View.

Table III **Alloy Separation and Metal Purification**[55]

Base Metal	Alloying Element %	Element in Condensate %	Element in Residue %	Temperature °C	Degree of Separation
Lead	0.51 Zn	—	0.037 Zn	603	good
	0.66 Sn	0.015 Sn	2.3 Sn	900	good
	0.92 Sb	0.11 Sb	0.99 Sb	800	fair
	0.63 Ag	0.002 Ag	0.64 Ag	800	good
	0.71 Cu	0.013 Cu	0.32 Cu	800	good
	0.65 As	0.10 As*	0.05 As	800	good
	0.37 Bi	0.18 Bi	0.38 Bi	800	poor
	50.0 Bi	52.1 Bi	39.06 Bi	800	poor
Tin	2.6 Pb	56.2 Pb	0.02 Pb	1000	good
	2.4 Sb	38.6 Sb	0.99 Sb	1100	fair
	2.0 Cu	0.5 Cu	2.0 Cu	1400	poor
	1.8 Fe	0.15 Fe	1.75 Fe	1400	fair
	1.5 Ag	7.03 Ag	0.12 Ag	1400	fair
	1.5 As	18.9 As	0.01 As	1400	good
Copper	3.0 S	4.64 S	0.94 S		poor
	18.69 Sb + 0.99 As	23.31 Sb + 0.78 As	4.1 Sb + 1.31 As		poor
	1.17 Sn	0.6 Sn	1.12 Sn		poor
	1.4 Sb	1.4 Sb	1.11 Sb		none
	saturated with O_2	0.25 O_2	0.84 O_2		poor

*Sampling losses.
For weight balances refer to original publication.

vacuum but the elimination of this metal can be more readily effected by overheating in a reverberatory furnace.

In one instance a vacuum arc furnace is employed for the treatment of cobalt-silver speiss. This seems quite logical. Lead, arsenic, antimony and sulphur can be eliminated with ease.[46] The treatment of matte might give similar results.

If the heating and corrosion problems inherent in such procedures could be solved, it would be a feasible proposition to apply vacuum in processes concerned with the recovery of gold contained in certain kinds of commercial antimony.

Dissociations and Reactions of Inorganic Compounds

The advantages offered by the provision of reduced pressures in the dissociation or reaction of inorganic compounds are the same as those pointed out above in connection with the distillation of metals, i.e. considerable reduction of temperatures and elimination of the danger from contamination by air. As a result clean reaction products are obtained. Compounds to be dissociated could be hydrides, oxides, nitrides, carbonates, sulphates and sulphites. Reaction products of such dissociations would be gases of the non-condensable type, as for instance, H_2, O_2, N_2, SO_2, SO_3 and CO_2. In passing, dehydration should also be mentioned. The dissociation pressures of various inorganic compounds are given in Table No. 4. It is doubtful whether a vacuum procedure would be economical in the case of the burning of lime, although the operational temperature could be lowered from approximately 897°C at 760 mm. Hg to 600°C at 1.8 mm. Hg and the CO_2 formed in the process could be collected in a state sufficiently pure for further use. But, where more expensive carbonates are concerned, for example Li_2CO_3 and $BaCO_3$, thermal dissociation *in vacuo* might be advisable if it is intended to obtain a pure oxide for use in metal winning by the Guntz method. The majority of oxides, except those of silver and mercury, have too low a dissociation pressure to suit production requirements. It is comparatively easy to dissociate sulphides[23], for example those of mercury, bismuth and antimony, but here again limitations are imposed in practice as the volatility of the metal may be too high and interfere with the reaction. However, it is possible to obtain tungsten and molybdenum by the dissociation of their respective sulphides.

At 1,000°C calcium sulphate has a dissociation pressure of 1.3 mm. Hg. This pressure can be increased to 11 mm. Hg by the addition of silica and to 15 mm. Hg by the addition of clay.[71] Potassium and sodium sulphate behave similarly but their dissociation pressures are of a higher order.

Another potential application of vacuum is in the dehydration of magnesium chloride followed by volatilisation at reduced pressure for the purpose of separating the oxide present. This method could be put into practice when the difficulties resulting from attack by the wet hydrochloric gas on the construction material have been overcome. The same applies to methods concerned with the reclamation of magnesium and the chlorides contained in waste magnesium foundry fluxes. Hydrochloric gas, in the dry state, will not damage mechanical vacuum pumps and steam ejector pumps are known to deal with such gases satisfactorily.

The large group of reactions which could be produced under vacuum for the purpose of obtaining inorganic compounds suitable for further metallurgical treatment has hardly been touched. Beryl ore can be reacted in vacuum at 1,000°C with the fluorides of aluminium or magnesium ; the beryllium fluoride is evaporated in the process.[24] The fluoride obtained can be reduced to beryllium metal with magnesium. Unfortunately the efficiency of this procedure is rather low. Similarly, clay reacts under vacuum with sodium fluoride yielding sodium aluminate and silicon fluoride. Aluminium fluoride and calcium aluminate are formed in a reaction with CaF_2.[79] True reactions and mere alterations in the proportional composition of compounds are both results of processes which have only recently been developed.[25,26,27] They provide for the production of lower aluminium halides (e.g. AlCl and AlF) at atmospheric pressure by dissolving aluminium scrap in the respective trihalides which in turn are

extracted as monohalides by vacuum distillation. On cooling, the monohalides yield aluminium metal and, in addition, trichloride or trifluoride which can be used for recycling. However, this process presents a number of technical difficulties which have not yet been overcome.

Another type of vacuum reaction deals with the reduction of a compound and the simultaneous formation of a volatile but easily condensable reaction product. Silicon forming the volatile silicon monoxide can be employed for the preparation of metals by reducing their oxides, i.e. Ta_2O_5 can be reduced in this manner to yield tantalum metal.[28,29] Silicon monoxide has a vapour pressure of 0.7 mm. Hg at 1,300°C.[81] The volatility of the oxides BO, AlO, SnO, GeO and Sb_2O_3 is higher than that of the corresponding oxides B_2O_3, Al_2O_3, SnO_2, GeO_2 and Sb_2O_4. This property of the former makes them suitable for reduction processes and could be utilised to effect their separation from alloys and slags. The lower sulphides of these metals, too, have a volatility which is higher than that of the corresponding normal sulphides and one could, therefore, separate tin from ore, for example, or other metals from mattes by sulphiding first. Subsequently the lower sulphides may be separated from any other sulphides present in vacuum. To quote a figure : the vapour pressure of SnS is about 1.8 mm Hg at 750°C.

WO_3 and MoO_3 which are fairly stable oxides could be separated from ores by heating in vacuum.

There are some roasting reactions, for example, those between Cu_2O and Cu_2S which could be performed at very low temperatures, if the pressure was reduced, but as mentioned before the reaction gas SO_2 would have to be pumped against atmospheric pressure and this might prove a very expensive procedure. The same applies to the reaction of carbon with oxides[11] and the group of reactions between carbon and sulphides which, according to the publications[30] quoted, yield carbon sulphide.

In many instances it is desirable to react oxides with chlorides. Where the chloride produced is volatile, it could be recovered without danger of oxidation from the air by providing a vacuum. Reactions of this type are sluggish at times and could be greatly assisted by adding other compounds which take part in the reaction (e.g. silica).

Roasting reactions in general embrace a wide range of chloride treatments carried out at a temperature level which facilitates the volatilisation of one of the reaction products. The following equation represents a typical reaction of this kind between iron oxide and calcium chloride assisted by the addition of silica as shown:

$$FeO + SiO_2 + CaCl_2 = FeCl_2 + CaO.SiO_2$$

Operating *in vacuo* and at a temperature of 1,000°C the iron dichloride is obtained in the gaseous state. In this manner anhydrous metal chlorides can be produced free of oxide since air is excluded ; if air were present at this temperature, it would react with the chlorides and liberate chlorine.One can substitute $MgCl_2$ or NaCl for $CaCl_2$ but then the reactions are less satisfactory due to the high volatility of these reagents. Calcium chloride is practically non-volatile, even at temperatures as high as 1,200°C. Silica can be replaced by other compounds (e.g. B_2O_3 or Al_2O_3) which bind the liberated base.

A switch of halogens may sometimes permit exchanging a less volatile chloride for a more volatile fluoride as demonstrated by the following reaction :

$$XF_2 + MgCl_2 = XCl_2 + MgF_2$$

Table IV **Dissociation Pressure of Various Inorganic Compounds.**

Mostly taken from Landolt Boernstein

Compound	Temperature °C	Pressure mm. Hg
$SrCO_3$	869	3.6
$BaCO_3$	824	8.6
$CaCO_3$	600	1.8
	650	6.9
	700	22.0
	897	760.0
Na_2CO_3	990	12.0
K_2CO_3	1300	15.1
Li_2CO_3	723	4.0
$MgSO_4$	950	24.0
$CaSO_4$	1000	1.3
FeO	1673	6.3×10^{-12}

All data in this Table are approximate only.

Fractional Distillation and Condensation of Inorganic Compounds

(See Tables No. 5 & 6)

It is frequently easier to separate metals from their compounds than from their alloys. This applies, in particular, to chlorides. The fractionation of halogen compounds by vacuum methods is barely mentioned in the literature. In some instances reference is made to the separation of chlorides of the cerium group, many of which have a vapour pressure of the order of about 1 mm. Hg in the temperature region of 400 to 475°C.[1] In certain cases it is possible to bind one element with a halide of low volatility such as fluorine, which will then be retained in the residue, while the more volatile chloride evaporates.

The main difficulties experienced in the fractionation of compounds originate from the low heat conduction properties of the solid charge and condensate. As pointed out before it is a serious problem to control the input of heat and its dissipation in the condenser effectively enough to make precision separation a practical possibility.

Chlorides can be reclaimed from oxide dross by fuming in a reverberatory furnace. In the course of refining tin alloys with lead oxide and tin chloride a tin oxide dross is formed which contains lead chloride.[48] In all probability, the lead chloride could be recovered with comparative ease if vacuum methods were employed.

The use of vacuum for the elimination of chlorides from metal sponge or from reduced metal particles is now common practice in the metallurgy of titanium and zirconium.[10] Separation techniques of this kind offer great advantages in the fusion electrolysis of chlorides which yield metal dendrites trapped in the electrolyte. They prevent the recovered electrolyte from being hydrolysed as happens frequently during extraction by wet methods. It may be that the sintering of the metal particles imposes certain limitations on the scope of this procedure. In the case of fluorides temperatures are required for their successful separation from metal sponges or crystals which prohibit the use of vacuum furnaces made of iron and of the type ordinarily employed in industry. As stated before, the maximum operational tempera-

Table V **Vapour Pressures of Some Inorganic Compounds.**

Mostly Taken from Landolt Boernstein

Compound	Temperature °C	Pressure mm. Hg
Al_2O_3	2360	6
As_2O_3	240.8	6.3
PbS	850	2.0
SiO_2	1900	23.0
SiO	1500	7.0
	1300	0.7
CdO	1028	2.1
PbO	1000	2.7
Sb_2O_3	600	2.2
SnO_2	1500	1.8
ZnO	1450	3.5
WO_3	1175	3.2
MoO_3	1155	760

All data in this Table are approximate only.

Table VI **Minimum Temperature at which Anhydrous Chlorides and Fluorides can be volatilised at an Industrial Rate (1.8 mm. Hg Pressure)**

Extrapolated from Landolt Boernstein

Chloride	°C at 1.8 mm.Hg
$AsCl_3$	—21
$BiCl_3$	20
$SbCl_3$	52
$AlCl_3$	129
$FeCl_3$	217
$ZrCl_4$	230
$CuCl_2$	295
$SnCl_2$	345
$ZnCl_2$	440
TlCl	445
$CoCl_2$	585
$PbCl_2$	590
CuCl	660
$CdCl_2$	700
KCl	710
NaCl	720
$NiCl_2$	740
$FeCl_2$	750
$MnCl_2$	770
$CsCl_2$	775
$MgCl_2$	805
LiCl	865
$CaCl_2$	nil at 1200
KF	1220
NaF	1330
LiF	1330
MgF_2	1510

All data in this Table are approximate only.

Vol. 1 1951

ture permissible for such furnaces is approximately 1,200°C.

In order to produce pure anhydrous manganese chloride one can react high carbon ferro-manganese powder with anhydrous ferrous or ferric chloride. The manganese chloride thus obtained can subsequently be separated from the iron powder by a distillation procedure *in vacuo*.[78] This method may prove to be cheaper than aqueous extraction of ores followed by a desiccation process and offers the additional advantage of yielding an oxide free chloride.

Production of Volatile Metals in a Vacuum

In 1906 Guntz proposed to reduce alkaline earth metals with aluminium in vacuum and actually produced barium and strontium in accordance with these proposals.[32] Later Matignon improved the method[33] and recommended the use of silicon in barium reduction. Matignon used silicon himself for the production of magnesium from calcined dolomite. He also reduced magnesium chloride with calcium carbide but failed to establish the process in industry because at that time—during the first World War—no heat resisting steels could be made available for the construction of retorts.[38,39]

Magnesium reduction by the silicon dolomite process was put into practice in Germany and Great Britain during the period 1938-1941, shortly afterwards in Australia[84] and finally in the United States in 1942. This process is based on the following reaction :—

$$Si + 2(CaO.MgO) = SiO_2.2CaO + 2Mg.$$

The operational temperature is 1,150°C and the vapour pressure immediately above the reaction mixture approximately 18 mm. Hg.[7] This method has been fully described elsewhere but reference to a few of the more important details of the process should be made in this article.

Pidgeon[7] employs retorts (see Figure No. 1) made from heat-resisting steel which are heated from the outside with gas or oil and have to withstand the pressure of the atmosphere at a temperature of about 1,150°C. They are of a comparatively small diameter and a great number has to be provided to allow for production on a large scale. As a result expenses for wages and maintenance are high, although the production cycle is of short duration.

Electro Metallurgical Company and Bagley[17] use the principle of internal heating and arranged for an electrical furnace to be placed inside an evacuated chamber (Figure No. 7). Their plant units are much larger than those of the Pidgeon type and their cycle of production is correspondingly longer but the expenses for wages are lower. On the other hand, the brick work of the furnace employed in the latter arrangement absorbs gases during each production cycle which are released in the course of the next cycle. This plant, therefore, is not suitable for the production of gas-sensitive metals, such as titanium or zirconium, but is adequate for the production of magnesium which does not dissolve either oxygen or nitrogen. Furthermore, before unloading can take place, it is necessary to cool down the charge to medium temperatures. This represents a considerable loss of heat not incurred in the operation of Pidgeon retorts, as the latter can be charged and discharged in the hot state. But the real objection to the use of a built-in electric furnace is its power consumption. 5 kWh are needed for the reduction of one pound of magnesium in Pidgeon retorts heated with oil. This is the energy required for the production of the ferro-silicon used in the process. In the case of the alternative process the power consumption of the electric resistance furnace has to be added to this figure raising the total power requirements to almost double the value mentioned for the Pidgeon equipment. If the fusion electrolysis method is used, a total of 9.2 kWh is required for every pound of magnesium produced. In other words the latter vacuum method has no advantages to offer compared with the atmospheric process. In fact, none of the vacuum methods is on a par with the method of fusion electrolysis as far as cost is concerned.[85]

De Coriolis[82] built a furnace providing for the heat—supplied by gas or oil burners—to be stored in the silicon carbide brickwork. After heating to the desired temperature, the furnace was evacuated and the charge introduced into the heat zone. This

furnace embodies many novel features but has only been used for a few heats.

If it is planned to provide internal heating by electrical means, it is preferable to allow for higher temperatures in an endeavour to shorten the production cycle and facilitate a reduction in the size of the furnace and the charge. Graphite resistor furnaces have been built for the reduction of magnesium[12] by the I. G. Farbenindustrie which operate at 1,300°C and a similar furnace is used in France for the reduction of calcium.[53]

Reducing calcium, it has to be taken into account that the vapour pressure over the reaction mixture of burned lime and aluminium is only of the order of 1 to 1.3 mm. Hg. The operational temperature is in the range of 1,150 to 1,200°C.[36] As higher temperatures are not permissible in retorts made from heat-resisting alloys, it takes a considerable time to produce sizeable quantities.[41] Also, the quality of the reduced metal is often impaired because retorts, especially the cast variety, are never really leak-proof and calcium dissolves nitrogen. If nitrogen is dissolved, a distillation process has to be added to secure final separation of the calcium.[83] The calcium carbonate, remaining in the burned lime, presents another difficulty. The heat penetrates at a very slow rate from the outside to the core of the charge and the removal of the residual carbon dioxide is not completed before the distillation of calcium begins in the hot region of the material. Indeed, the two effects overlap and calcium vapours reaching the condenser react with CO_2, as a result of which the calcium is contaminated with carbon. The presence of carbon and, especially nitrogen, is not desirable in cases where the calcium is to be used for the reduction of titanium dioxide. Nitrogen hardens titanium to a much greater degree than oxygen. Another impurity usually contained in the metal is magnesium. In spite of these difficulties calcium is produced by this process on an industrial scale and its quality is superior to that of the electrolytic product which contains large amounts of chloride and must normally be remelted for most applications. It is claimed that calcium can be produced in retorts at less than 1 dollar per pound. If the operational temperature could be raised, the time of the production cycle and the size of the furnace could be reduced. Here, the use of graphite-rod resistor vacuum furnaces[12,53,88] which operate at 1,300°C instead of retorts should greatly benefit the method (see Figure No. 5). Details of the construction and performance of these furnaces are not available.

Barium has been produced for a great number of years[33,51] by the reduction of BaO with aluminium. The vapour pressure of the metal above the reaction mixture, 3 BaO + 2 Al, can be assumed to be low as the operational temperature is restricted to about 1,200°C. Even at 1,400°C the metal is estimated to have a vapour pressure of only 1.8 mm. Hg. The furnace designed for the process by the I.G. Farbenindustrie has been described elsewhere.[86] Condensation of the metal takes place in liquid form under a protective gas atmosphere and ingots are cast directly from the condensate. This procedure constitutes an appreciable advance, since barium is a very active metal and heats up when exposed to air due to nitriding and oxidation, in certain conditions even catching fire. In the form of castings barium is much less sensitive. Barium could propably be produced at an expense of less than 1 dollar per pound, if the demand would justify large-scale production. As matters stand today it is mainly used as a getter in certain applications, particularly at low temperatures, where other getters are not effective.

Lithium can be produced in a vacuum by reducing a mixture of lithium carbonates and burned lime previously degassed *in vacuo* with aluminium at about 1,150°C.[34,35] It has also been attempted to reduce spodumene with a 0.23% lithium content by high grade ferro-silicon at 1,150°C and an addition of burned lime.[34,35] Lithium was recovered at a rate of about 90%. The charge consisted of 67.3 parts of burned lime for every 28.8 parts of spodumene and an addition of 3.9% FeSi as the reducing agent. Judging by the volume of the retort one can see that the rate of recovery is low. In addition, the condensate obtained contains a great number of impurities i.e. sodium, potassium and magnesium. All these materials would have to be separated from the lithium by fractionation. The product is pyrophoric

Vol. 1 1951

and, whilst it may be possible to manipulate it under light oils, the extra cost and difficulties inherent in this procedure do not favour the adoption of the process. In fact, it is much easier to eliminate the bulk of the ore by aqueous methods, to precipitate the lithium from the solution and to continue the treatment on the lines as described above.

Many other oxides of volatile metals, as for instance, those of lead, zinc, manganese and bismuth, could be reduced by silicon in vacuum but the conventional methods are cheaper.

In the course of the reduction of dolomite in vacuum a by-reaction takes place which yields sodium and has quite a disturbing influence on the process. One can reduce sodium chloride with silicon in the presence of lime[37] as follows :—

$$4\,NaCl + Si + 4\,CaO = SiO_2.2\,CaO + 2CaCl_2 + 4\,Na$$

The process requires a temperature of about 800°C. Therefore, iron retorts with inconel sheathing can be employed. Production on a large scale has been carried out for some time in accordance with this method. It needs little equipment and obviates the necessity of dealing with chlorine as a by-product. It could be applied in areas, where the supply of electrical power is short and the demand for chlorine negligible.

Cæsium and Rubidium can be reduced from their chlorides under vacuum in small quantities. Calcium is used as a reagent. The apparatus should be made from glass.

The I.G. Farbenindustrie is producing potassium by a method outlined in the following equations[40]:

1. $2\,KF + CaC_2 = CaF_2 + 2\,C + 2\,K.$
2. $Si + 4\,CaO + 4\,KF = 2\,CaF_2 + SiO_2.2\,CaO + 4\,K.$

The rate of recovery is 80 to 90%. Due to the high temperatures (1,100—1,500°C) required in the process the iron retorts do not last for more than about 10 production cycles. In the case of the silicon reduction following equation No. 2, potassium carbonate-fluoride was employed as an alternative but this resulted in explosions. It is evident that this method could be greatly improved by operating *in vacuo*. The operational temperatures would be lower and the iron of the retorts could be adequately protected. No such equipment has yet been developed.

The use of calcium carbide for chloride and fluoride reductions is often suggested.[38,39,70] The materials concerned are the chlorides of volatile metals such as lead, zinc and the alkali metals. The reduction of NaCl and KCl in vacuum is frequently mentioned in the literature.[72,73,39] In the case of NaCl recoveries of the order of 80% are obtained.[70]

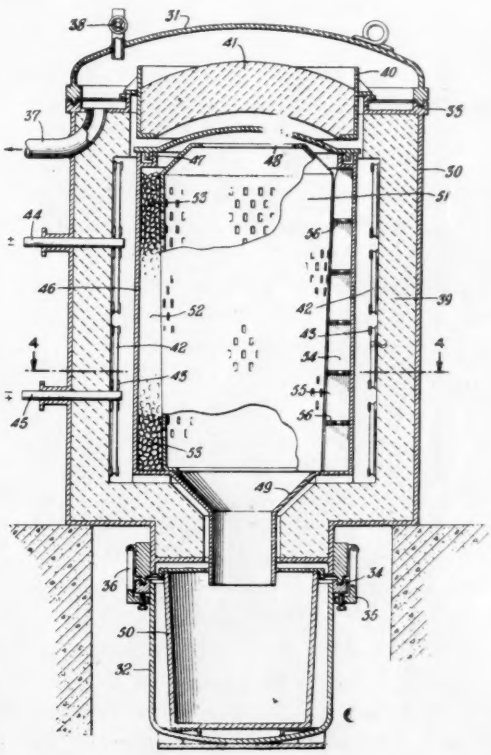


Fig. 7. Bagley-Union Carbide vacuum furnace for producing magnesium from dolomite.[17]

Description of numbers :
30. Outer shell. 31. Outer cover. 32. Outer condenser shell. 33. Rubber gasket. 34. Rubber gasket for condenser. 35. Clamp. 36. Clamp. 37. Vacuum outlet. 38. Air inlet. 39. Refractory and heat insulation. 40. Inner cover. 41. Refractory and insulation. 42. Heater elements. 43. Element holder. 44 and 45. Electrode inlets. 46. Inner charging drum. 47. Seal. 48. Radiation screen and cover. 49. Funnel. 50. Inner condenser. 51. Perforated inner funnel. 52. Annular charge space. 53. Batch. 54 and 55. Individual sections. 56. Gas escape holes.

Calcium carbide, besides being expensive, is contaminated to about 50% with non-reacting materials, mainly carbon and oxide. In addition at higher temperatures, reacting with moisture which condenses on its surface, it often produces acetylene. Similar objections apply to the use of CaC_2 for the reduction of various oxides and sulphides[70], but the reduction of zinc silicates with CaC_2 has been recommended. The peculiar properties of calcium carbide should assist in this reaction.

It has been repeatedly proposed to carry out sulphide reductions in vacuum. Zinc sulphide can be reduced with finely divided iron[23,75] according to :—

$$Fe + ZnS = FeS + Zn$$

Due to the fact that the operational temperature is as high as 1,000°C a certain amount of zinc sulphide volatilises at the same time. Therefore, condensation presents certain difficulties in this process. Iron could be readily obtained by reducing iron ore with producer gas which need not be particularly pure for the purpose but there is little to be gained from this method. No proof has been given yet that lead and cadmium can be condensed selectively in order to obtain high purity zinc. At all events, the residue would have to be roasted in air to recover sulphur. Any noble metals present would be lost in the residue, unless the residue were used over and over again and ultimately treated for its metal content.

Various other metal sulphides, e.g. those of lead and mercury, could be reduced with iron *in vacuo* but none of these suggestions has materialised into an accepted practical procedure.

CONCLUSIONS

This study mentions a great number of possible applications of vacuum in metallurgy but only a few of these are firmly anchored in industry. The silicon-dolomite process was almost completely abandoned after the last war. Only one firm continued operations in accordance with this method relying on the excellent purity of the product for its commercial success. However, interest in this method is reviving just now. The separation of magnesium chloride from titanium and zirconium sponge in retorts at reduced pressure is well established. Calcium, barium and strontium are produced by the Guntz process with some difficulties. The elimination of zinc from lead under vacuum after de-silverisation and the separation of speiss in an arc vacuum furnace are carried out regularly in industry today. Finally, it is an accepted practice to separate magnesium-aluminium alloys obtained in refining aluminium scrap by the Beck method in vacuum furnaces.

Applications which have not yet found their way into production proper but hold great promise of success are : the melting, de-gassing, purification and decarburisation of stainless heat-resisting and silicon transformer steels, the decarburisation, with various oxides, of ferro-chromium in the solid or fused state, the partial or complete elimination of lead from tin alloys and the de-zincing of crusts formed in the course of de-silverisation procedures.

There is a plausible explanation for the fact that vacuum methods are not used yet on a more universal scale. High capacity oil diffusion pumps producing very low pressures have only been available for the last 10 years. The same applies to vacuum valves and reliable measuring instruments. Another restriction is the fact that vacuum processes must be carried out in batches. Only a few metals melt readily and can be fed to or discharged from a vacuum furnace continuously by means of barometric columns. Attempts have been made to develop continuous vacuum processes[87] but the design of large vacuum valves required for such equipment raises a number of difficult problems. Also, the heat transfer to a stationery solid batch of material under vacuum is slow, except where arc- or high-frequency furnaces are employed, whereas at atmospheric pressure the gases in blast or reverberatory furnaces assist heat transmission greatly. Consequently, vacuum equipment is bulky and expensive in relation to its output capacity.

Other limiting factors are the inadequacies of the available plant materials. Their creep strength is insufficient and their resistance to oxidation from the air and chemical attack by the charge is too low.

Energetic steps have been taken to deal with these problems. For example the recent introduction in

Vol. 1 1951

industry of the graphite resistor furnace of the rod type extended the range of operational temperatures to 1,300°C. High-frequency furnaces, very useful for high temperature melting work, will have to be re-designed, before they can be applied to the distillation of metals. The processing of non-conducting material in such furnaces is another problem still largely unsolved.

Research work on the properties of plant materials is greatly needed, especially on their behaviour, when exposed to the vapours of metals and inorganic compounds. This applies, in particular, to the stability of iron and the dissociation reactions of its compounds on contacting zinc, arsenic, antimony, sulphur, selenium, tellurium and manganese. Eventually iron alloys will have to be made available which are completely unaffected by these conditions. Finally, the resistance of refractories to hot chlorides, fluorides, oxides and sulphides in vacuum should be investigated. Summing up, it can be said that furnace design is bound to benefit considerably from any results of basic research on these problems.

It is certain that we are in the initial stage of great developments which in course of time will reach and enter branches of metallurgy not even thought of at present. The application of vacuum may be a costly matter but it is a first-rate means of ensuring precision and refinement in processing. In the course of time it will firmly entrench itself in the front line of a steadily advancing metallurgy.

References

1 KROLL, W. J., *Trans. Electrochem. Soc.*, **87**, (1945), 571-587.
2 ISBELL, W. T., U.S. Patent, 2,422,967.
3 ISBELL, W. T., *Amer. Inst. Min. Metall. Engrs., Met. Technol.*, **14**, (No. 3, 1947), Tech. Paper No. 2138.
4 HOCHMAN, J., *Rev. Metall.*, **44**, (1947), 161-173.
5 HOCHMAN, J., *C. R. Acad. Sci., Paris*, **226**, (1948), 2150-2151.
6 HOCHMAN, J., *Metal Progr.*, **59**, (No. 6, 1951), 852.
7 PIDGEON, M. L., and ALEXANDER, W. A., *Trans. A.I.M.E.*, **159**, (1944), 315-352.
8 MAYER, A., *Trans. A.I.M.E.*, **159**, (1944), 363-376.
9 ENSIGN, E. E., and SCHNAUB, F. G., U.S. Patent, 2,390,698.
10 KROLL, W. J., HOLMES, H. P., YERKES, L. A., and GILBERT, H. L., *Trans. Electrochem. Soc.*, **94**, (1948), 1-20.
11 KROLL, W. J., and SCHLECHTEN, A. W., *Trans. Electrochem. Soc.*, **93**, (1948), 247-258.
12 BALL, C. J., FLETCHER, S. J., and FOX, F. A., *B.I.O.S. Final Report No.* 1338, Item 21.
13 KROLL, W. J., ANDERSON, C. T., and GILBERT, H. L., *Trans. A.I.M.E.*, **175**, (1948), 766-773.
14 KROLL, W. J., and GILBERT, H. L., *Trans. Electrochem. Soc.*, **96**, (1949), 158-169.
15 STAUFFER, K. F. R. A., and DI PIETRO, W. O., *Iron Age*, **161**, (No. 9, 1948), 79-84.
16 KROLL, W. J., *Trans. Electrochem. Soc.*, **79**, (1941), 199-213.
17 BAGLEY, G. D., U.S. Patent, 2,386, 189.
18 PARKE, R. M., and HAM, J. L., *Trans. A.I.M.E.*, **171**, (1947), 416-430.
19 HUTTON, J. G., *Gen. Elect. Rev.*, **52**, (No. 10, 1949), 12.
20 JACOBS, H., and LA ROCQUE, A. P., *Phys. Rev.*, **73**, (No. 10, 1948), 1244.
21 ESPE, W., and KNOLL, M., *Werkstoffe der Hochvakuum Technik*, (Springer Verlag, Berlin, 1936), 234.
22 BURKHARDT, W., and SCHICKER, H., German Patent 739,974.
23 ELLINGHAM, H. J. T., *J. Soc. Chem. Ind.*, **63**, (May 1944), 125-133.
24 WILMORE, C. B., and CHEW, F. D., U.S. Patents 2,387,203 ; 2,387,204 ; 2,387,205 ; 2,387,207.
25 GROSS, P., U.S. Patents 2,470,305 ; 2,470,306.
26 KLEMM, W., VOSS, E., and GEISBERGER, K., *Z. Anorg. Chem.*, **256**, (1948), 15-24.
27 KLEMM, W., and VOSS, E., *Z. Anorg. Chem.*, **251**, (1943), 233-240.
28 ZINTL, E., *Z. Anorg. Chem.*, **245**, (1940), 1-7.
29 ZINTL, E., *Z. Anorg. Chem.*, **245**, (1940), 8-11.
30 FRAENKEL, W., *Metallurgia*, **6**, (1909), 682.
31 VICKERY, R. C., *J. Soc. Chem. Ind.*, **65**, (1946), 388-390.
32 GUNTZ, A., *C. R. Acad. Sci., Paris*, **143**, (1906), 339-340.
33 MATIGNON, C , *C. R. Acad. Sci., Paris*, **156**, (1913), 1378-1380.
34 KROLL, W. J., and SCHLECHTEN, A. W., *Trans. A.I.M.E.*, **182**, (1949), 266-274.
35 STAUFFER, R. A., *Trans. A.I.M.E.*, **182**, (1949), 275-285.
36 ATKINSON, J. T. N., and PIDGEON, L. M., *Trans. Canad. Inst. Min. Met.*, **51**, (1948), 26-32.
37 KROLL, W. J., U.S. Patent 2,465,730.
38 MATIGNON, C., *C. R. Acad. Sci., Paris*, **172**, (1921), 381.
39 GEL'D, P. V., and MIKULINSKIJ, A. S., *J. Appl. Chem., U.S.S.R.*, **20**, (1947), 800-808.
40 SMATKO, J. S., *F.I.A.T. Final Report No.* 695.
41 LOOMIS, C. C., *Trans. Electrochem. Soc.*, **89**, (1946).
42 LEITGEBEL, W., *Metall. und Erz*, **32**, (1935), 205-211.
43 GRUBE, G., and SCHNEIDER, A., *Z. Anorg. Chem.*, **260**, (1949), 120-126.
44 ST. CLAIR, A. W., and SPENDLOVE, M. J., *U.S. Bur. Min., Rep. Invest.* 4710, (1950).
45 SCHLECHTEN, A. W., *Engng. Min. J.*, **150**, (No. 12, 1949), 80-81.
46 ANON., *Iron Age*, **164**, (December 1, 1949), 114-116.

47 OSBORN, W. H., and TWINNER, S. B., U.S. Patents 2,239,370; 2,239,371.
49 BEHR, G. E., *Amer. Inst. Min. Metall. Engrs., Met. Technol*, **10,** (October, 1943), 56-65
49 PIDGEON, L. M., and KING, J. A., *Trans. Faraday Soc. Discussion*, (No. 4, 1948), 197-206.
50 KUBASCHEWSKI, O., *Trans. Faraday Soc., Discussion*, (No. 4, 1948), 241.
51 KROLL, W. J., *U.S. Bur. Min., Inform. Circ.*, **7326,** (1945).
52 PIDGEON, L. M., Canad. Patents 415,764; 415,765.
53 EICHNER, C., GOLDSCHMIDT, B., and VERTES, P., *Bull. Soc. Chim. Fr.*, (January-February, 1951), 140-143.
54 DAVENPORT, W. H., *J. Amer. Ceram. Soc.*, **33,** (No. 11, 1950).
55 ERASMUS, H. W., U.S. Patents 2,473,019; 2,473,020; 2,473,021.
56 SPENDELOW, H. R., KENMORE, J., and ERASMUS, H. W., U.S. Patent 2,473,021.
57 CHADWICK, C. G., U.S. Patent 2,541,153.
58 BLACKWELL, H. A., and TURNER, W. L., Brit. Patent, 269,760, and Brit. Patent, 466,763.
59 SCHNEIDER, A., and ESCHE, U., *Z. Elektrochem.*, **45,** (1939), 888-893.
60 SCHNEIDER, A., and STOLL, E. K., *Z. Elektrochem.*, **47,** (1941), 519-526.
61 SCHNEIDER, A., and STOLL, E. K., *Z. Elektrochem*, **47,** (1941), 527-535.
62 SCHNEIDER, A., and SCHMIDT, H., *Z. Elektrochem*, **48,** (1942), 627-639.
63 SCHNEIDER, A., and SCHMIDT, H., *Z. Elektrochem*, **48,** (1942), 640-646.
64 KUBASCHEWSKI, O., *Z. Elektrochem*, **48,** (1942), 559-582, 646-659.
65 LOEWENSTEIN, H., U.S. Patent 2,198,673.
66 FOUNDAMINSKI, T. J., and LOEWENSTEIN, H., French Patent 848,375.
67 ST. CLAIR, A. W., and BLUE, D. D., *U.S. Bur. Min. Rep. Invest.*, **4535,** (1949).
68 ANON., *Metal Ind., Lond.*, **69,** (1946), 235-238.
69 POLAND, F. F., *Amer. Inst. Min. Metall. Engrs., Met. Technol.*, **13,** (September, 1946), Tech. Publication No. 2065.
70 JUNGFLEISCH, M. L., *Ann. Chim.*, **2,** (Series 12, 1947), 129-157.
71 BRINER, E., PAMM, G., and PAILLARD, H., *Helv. Chim. Acta*, **31,** (1948), 2220-2235.
72 HACKSPILL, L., and STAEHLING, C., German Patent 363,735.
73 FREEMAN, H., U.S. Patent 1,319,148.
74 ACKER, C. E., *Trans. Elektrochem. Soc.*, **1,** (1902), 165.
75 GROSS, P., and WARRINGTON, M. M., *Trans. Faraday Soc., Discussion*, (No. 4, 1948), 215-217.
76 KROLL, W., *Metal Ind., Lond.*, **47,** (1935), 3-6, 29-31, 103-105, 155-156.
77 MOTOCK, G. T., *Iron Age*, **158,** (December 12, 1946) 64-69.
78 KROLL, W. J., and BACON, F. E., U.S. Patent 2,452,665.
79 MOROZOV, G. S., and IVANOV, N. A., Russian Patent 52,597.
80 VON WARTENBERG, H., *Z. Elektrochem.*, **20,** (1914), 443-449.
81 VON WARTENBERG, H., *Z. Elektrochem.*, **53,** (1949), 343-347.
82 DE CORIOLIS, E. G., *Trans. A.I.M.E.*, **182,** (1949), 93-112.
83 BETCHERMAN, I. I., and PIDGEON, L. M., *Canad. Min. Metall. Bull.*, **44,** (No. 468), 253-263.
84 KEMMER, F. R., *Trans. Electrochem. Soc.*, **81,** (1942), 65-88.
85 KLAGSBRUNN, H. A., *Industr. Engng. Chem.*, **37,** (1945), 608-617.
86 YOUNG, K. W., C.I.O.S. Report III-31, Item 22.
87 PRYTERCH, W. E., Brit. Patient, 545,312.
88 ANON., *Mod. Metals*, (March, 1951), 54-55.

An introduction to HIGH VACUA IN NUCLEAR PHYSICS

A. S. Baxter, M.Sc., Ph.D. Cavendish Laboratory, University of Cambridge.*

Summary

THE NEED for high vacua in the study of nuclear physics and the consequent parallel development of these subjects is outlined. The nature of nuclear reactions and the vacuum requirements of associated instruments is then described.

Sommaire

L'AUTEUR EXPOSE le developpement qu'a pris la technique du haut vide parallelement avec l'étude de la physique nucleaire, laquelle en est tributaire. La nature des réactions nucleaires et les exigeances quant au vide d'instruments employés à cet effet sont ensuite décrites.

ol.
1
951

INTRODUCTION

WHEN RUTHERFORD predicted in 1914 that the nucleus of an atom might be altered by direct collision with very swift atoms of helium (alpha-particles) it is probable that he already had in mind the experiment which he finally carried out in 1919 and by which he became the first to demonstrate the artificial transmutation of an element. Transmutation has become the most important method of nuclear research ; it is also the one which has relied most on vacuum technique for its successful accomplishment.

Since efficient pumps are the foundation of any vacuum system, vacuum technology may also be said to have started about this date with the invention of the mercury pump by Gaede in 1915—an invention which was quickly followed by the introduction in 1916 of the condensation principle of Langmuir. The years 1919-1926 saw the confirmation and extension of Rutherford's work and laid the foundation of the 'collision–disintegrative' method of nuclear study. But the need was soon felt for a more controllable source of high energy particles than the naturally occurring alpha-particles used at that time. By 1928 several laboratories were engaged in the construction of equipment thought suitable for this purpose and in 1932 the first nuclear reaction carried out by artificial means was performed by Cockcroft and Walton in the Cavendish Laboratory. Their success was quickly followed by the introduction of other methods for the acceleration of particles to high energies – methods which would have been quite impossible without the adequate provision of high vacuum.

* M.S. Received July, 1951.

Editor's Note : This is an introductory article to a series appearing in future issues of 'Vacuum', dealing with vacuum techniques as they affect the design and performance of nucleonic plant.

NUCLEAR REACTIONS

The atomic constitution of matter is now well established. The atom itself has been shown to possess structure and to consist of a small massive positively charged nucleus surrounded by a cloud of negatively charged electrons such that the atom as a whole is electrically neutral. The arrangement of these orbital electrons is responsible for the chemical nature of the atom so that isotopes – atoms whose nuclei bear similar charges but different masses – are indistinguishable chemically.

The present concept of nuclear structure attributes

to the nucleus most of the atomic mass and a composition of positively charged protons together with neutrons of equal mass but zero charge. Every nucleus is thus characterised by the number of neutrons and protons it contains. Every nuclear transformation, therefore, implies a change in this number, either of protons or of neutrons or both. Penetration into the nucleus is, therefore, a prime requirement for a nuclear reaction, and whatever agency is employed must have sufficient energy to effect such a penetration. The result could be simply the addition of another particle to the nucleus or, more generally, the ejection of some of those already there. These ejected particles form, together with the residual nucleus, the disintegration products of the reaction.

That the positively charged protons in the nucleus are not themselves ejected by mutual repulsion (Coulomb's law) is explained by the assumption of an extremely powerful nuclear force which only becomes effective when elementary particles approach to within distances of the order of nuclear dimensions. Quite apart from the intrinsic interest of nuclear transformation, an understanding of the nature of these forces is, in fact, one of the fundamental aims of nuclear research.

The course of a typical reaction is shown diagrammatically in Fig. 1. A beam of accelerated particles is directed against a target containing the desired nuclei, any successful encounter leading to the appearance of disintegration products.

PARTICLE ACCELERATORS

Considerable incentive was given to the initial development of particle accelerators by the theoretical studies of Gamow in 1928. Gamow showed that the probability of nuclear penetration was increased as the charge and mass of the bombarding particle decreased. For a given value of particle energy, therefore, the proton (hydrogen nucleus) with unit charge and unit mass was more likely to be effective than the alpha-particle with a charge of two and a mass of four units.

In order to accelerate a particle by electrical means, it must carry an electric charge. The simplest method of doing this is clearly to remove one of the attendant electrons and thus leave a positively charged atom. Acceleration is then simple – at least in principle. It is merely necessary to allow the ionised atom to fall through a sufficient potential difference. In this manner the electron volt (ev) as a unit of energy becomes a natural measure, for it is the energy received by a charge of unit magnitude in falling through a potential of one volt. In terms of this unit, the alpha-particles used by Rutherford in his original experiment possessed an energy corresponding to a potential difference of 7.7 million volts, i.e. an energy of 7.7 Mev. But even this very high value was insufficient to enable Rutherford to effect transmutation with elements higher than potassium in the periodic table. Light particles (protons) at higher energies were needed and were to be provided by the particle accelerators, then under development.

Particle accelerators may be divided essentially into two fundamental types, in the first of which the energy required is reached in one step and in the second attained by repeated applications of much smaller steps. An alternative description could be based on the physical principles employed, i.e. whether electrostatic or electro-magnetic (radio-frequency) methods are involved. Examples of the former class are the electrostatic generator of Van De Graaff and the transformer rectifier system used by Cockcroft and Walton. Both supply a steady constant potential, the Van De Graaff machine particularly so. In both these instruments acceleration is accomplished by guiding the particles, derived by ionisation of an appropriate gas, along an evacuated acceleration tube, across the ends of which the full potential of the high voltage terminal is applied. Machines such as this, although of comparatively small energy, are unique in the beam intensities which they provide with little energy spread. Both these forms of generator have been developed to the stage where they are commercially available.

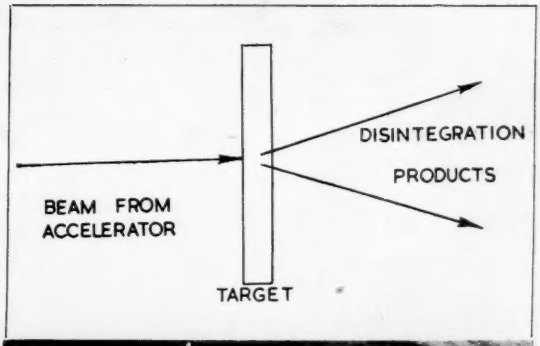
The first instrument utilising radio-frequency potentials was the linear accelerator of Lawrence and Sloan (1931). Here, the particle was accelerated across the gaps between a number of linearly mounted

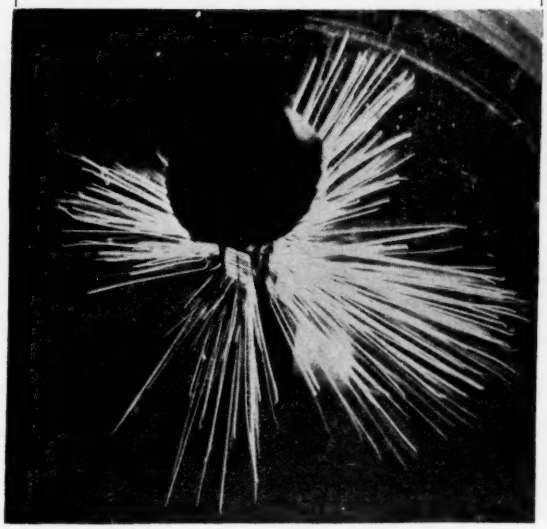
Vol. 1 1951

cylinders with alternate cylinders connected to one terminal of a radio-frequency supply. It is possible to choose the frequency so that the field between the gaps is always in a direction to accelerate the particle into the following cylinder, but the lack of suitable radio-frequency oscillators prevented the full application of this method at that time. The development during the recent war of the magnetron valve, in itself a considerable achievement of high vacuum engineering, has changed this picture and the linear accelerator is again under vigorous development.

The cyclotron of Lawrence (1931), perhaps the most famous of all nuclear machines, although similar in principle is radically different in form. The shielding cylinders of the linear accelerator are replaced by two flat D-shaped boxes placed between the poles of a magnet. The action of the magnetic field constrains the particle to move in a spiral of increasing radius as it gains energy by repeated traversals of the gap between the D's. Thus the length of the path automatically increases with the speed of the accelerated particle. There can be little doubt that much of the present standard of high vacuum engineering is due to the effort expended in the development of these machines.

With constant fields and potentials the maximum energy obtainable is limited by the strength and size of the magnetic field in which the ions traverse their spiral path. But even if economic considerations permitted the construction of the enormous magnets required for very high energies, the relativistic increase of mass would prevent the successful operation of the conventional cyclotron principle without modification.





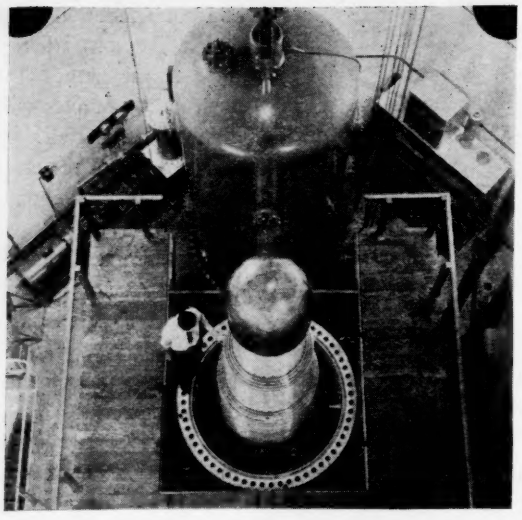
Cavendish Laboratory

Fig. 1. *Above:* Diagram showing the sequence of events in nuclear reactions. *Below:* Cloud chamber photograph of disintegration products (alpha-particles) from the proton bombardment of a lithium target.

Recently, two such modifications have been introduced. By changing the radio-frequency supply in synchronism with the increasing particle energy whilst maintaining a constant magnetic field, it has been found possible to produce 350 Mev protons from the 184-inch diameter synchro-cyclotron at the University of California.

Proton synchrotrons using both varying radio-frequency and pulsed magnetic fields are under construction in a number of laboratories. With these it is hoped to reach proton energies exceeding 1,000 Mev. At such energies the particles approach a substantial fraction of the velocity of light and, since the acceleration time may be as long as one second, the distance travelled during acceleration may exceed a hundred thousand miles.

It is significant that each increase in energy in the past has lead to the discovery of important new facts, and there is every reason to believe that this will again be so, when energies of the order of 1,000 Mev and greater are achieved.



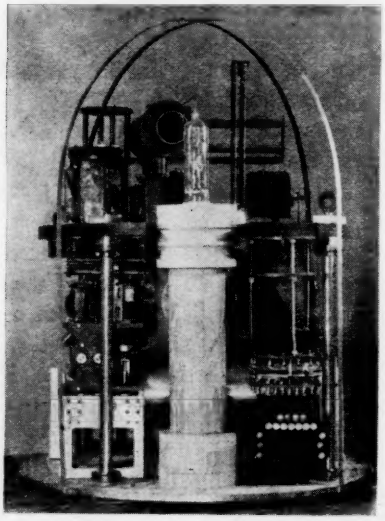
Cavendish Laboratory

Fig. 2. 4 Mev electrostatic generator. Pressure tank removed.

VACUUM REQUIREMENTS

In electrostatic accelerators the concept of 'mean free path' is of great assistance in visualising the necessary vacuum conditions. By mean free path is meant the average distance between collisions in a gas at a given pressure. At atmospheric pressure any gas, irrespective of its nature, contains about 2.7×10^{19} molecules per cubic centimetre and, since a molecule of a gas may contain anything from one to ten atoms, this leads to a value of approximately 10^{11} atoms per cubic centimetre, if the pressure is reduced to 10^{-6} millimetres of mercury. The fact that the average distance between collisions is of the order of 50 metres under these conditions is a clear illustration of the very small values of atomic dimensions. The avoidance of collisions in such accelerators is thus a question of sufficiently reducing the gas pressure, until the mean free path becomes of the same order as the acceleration length. That collisions are undesirable is obvious for several reasons, quite apart from scattering and resultant loss of beam intensity. Ionisation in the acceleration tube as a result of such collisions may result in back bombardment of the ion source by the electrons so formed, thus unintentionally converting the instrument into a high voltage X-ray machine. The health hazards thus created may render additional shielding necessary.

The acceleration tube undoubtedly presents the most difficult vacuum problem, particularly on machines designed for high energy. Considerable uncertainty exists about the fundamental mechanism of electrical breakdown over insulated surfaces in vacua – an uncertainty which is reflected in the wide variety of tube designs published to date. In order to prevent electrical breakdown over the external surface it has become common to use compressed gas insulation in modern compact generators because an increase in the dielectric strength of the atmosphere surrounding the electrode system allows the physical dimensions to be correspondingly reduced without at the same time lowering the breakdown voltage. In order to ensure a uniform distribution of potential down the accelerating tube, the latter is generally sub-divided by a series of electrodes separated by short insulating spacers, and the vacuum seals at the junctions of such sections must, therefore, be



Cavendish Laboratory

Fig. 3. Ion source and energising equipment of the electrostatic generator.

Vol. 1 1951

subjected to the external pressure of the compressed gas insulation. With the many seals required, this imposes a stringent condition, the more so when it is considered that up to a thousand such seals may be necessary.

Unfortunately the ion source gives rise to an appreciable flow of neutral gas which must be quickly removed to prevent a build-up of pressure. On the other hand, the pumping speed of the tube is limited by the physical size and necessary mechanical strength of the system so that, in practice, these considerations generally lead to an operating potential often 50% less than that obtainable in the absence of an ion beam. Fig. 2 shows the Cavendish Van De Graaff generator with the pressure tank removed, and Fig. 3 its ion source and energising equipment.

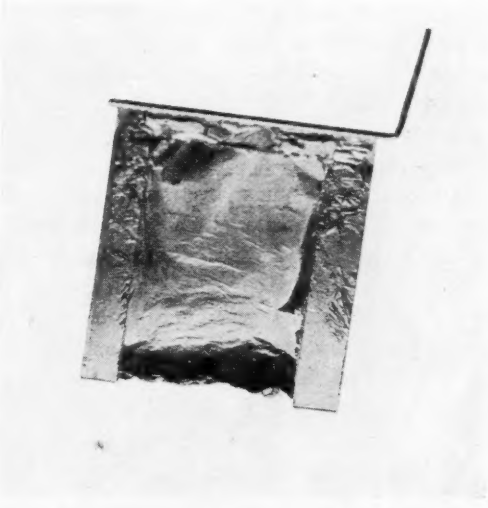
In electro-magnetic instruments where the accelerated particle may travel great distances before emergence, the concept of mean free path alone is of little use, for it is clearly quite impracticable to reach values even approaching the acceleration length. The physical dimensions are generally such that provision of adequate pumping speed is not difficult even when the somewhat generous allowance of one litre per second per litre of evacuated volume is allowed. One is rather more concerned that the minimum ultimate pressure shall arise from low molecular weight constituents with a view to reducing the adverse effects of the inevitable collisions to the smallest possible value.

TARGET PREPARATION

The efficiency of nuclear reactions in terms of successful disintegrations per bombarding particle is generally very small – frequently of the order of one for every million incident particles. The rest merely lose their energy by interaction with the extra-nuclear electrons, through which they must pass before they can approach sufficiently close to a nucleus to have a chance of penetration. Thus the process as a whole is very inefficient because of the large number of bombarding particles which do not produce any reaction. With the beam intensity limited by other considerations, it is obviously desirable to use as target material a compound containing as many desired nuclei as possible. Chemical compounds, however, consist of naturally occurring isotopic mixtures and thus isotope separation becomes an important subject. A target consisting largely of a single isotope has, in addition to the higher proportionate yield, the further distinct advantage that possible competing reactions are considerably reduced in number. The development of electro-magnetic separation by essentially mass spectrometer techniques has progressed remarkably in the last ten years, and surprisingly large amounts of many rare isotopes are now available.

For very thin targets vacuum evaporation techniques are indispensable. Frequently such targets are required free from any substrate. A typical example is the free beryllium film shown in Fig. 4.

Target contamination by decomposition of back-streaming oil vapour from the diffusion pumps is of frequent occurrence. Carbonaceous contamination is not always serious, although it can occasionally give rise to subsidiary reactions.



Cavendish Laboratory

Fig. 4. Self-supporting beryllium target. Area : 2 × 1½″. Thickness : 900 Å.

DETECTION OF DISINTEGRATION PRODUCTS

Ionisation in a gas is frequently used to identify and record the radiation passing through it. In the case of charged particles, the ionisation is produced by the direct action of their electric fields on the orbital electrons of the gas atoms. Neutrons and gamma rays, on the other hand, do not ionise directly and must first be persuaded to convey their energy to a charged particle by an intermediate process before they can be detected. The development of the ionisation method has yielded a wide range of instruments from the relatively low pressure Geiger counter to the high pressure ionisation chamber containing gas at several atmospheres. The vacuum techniques employed throughout their manufacture are those of the electron tube industry, i.e. of static vacuum systems. The procedures are in many ways similar and impose the same limitations on materials and techniques.

The detection of nuclear radiations by the visible light they produce in certain fluorescent materials has had an interesting history. The famous scattering experiments of Geiger and Marsden which confirmed the Rutherford concept of the nuclear atom, the transmutation experiments of Rutherford and the first artificial reaction of Cockcroft and Walton, all used this method. The light output (scintillations) produced by the impact of an energetic alpha-particle or proton on a screen of zinc sulphide is easily visible to the human eye but, while the latter remained the only detector available, the method was necessarily slow. In addition, the human eye had a regrettable tendency to see flashes which did not exist.

By 1935 the vacuum techniques of electron tube manufacture had reached the stage where it became possible to utilise, in a practical manner, the principle of secondary electron multiplication proposed by Slepian in 1919. Electrons originating at the first of a series of secondary emitting surfaces liberate a larger number of secondary electrons at each succeeding stage so that, with sufficient stages, exceedingly high amplification is possible.

The provision of a photo-cathode as the initial surface – yielding electrons under the stimulus of light – converts the electron multiplier into a photo-electron multiplier. Subsequent development has produced photo-multipliers capable of detecting the scintillations produced when phosphors are irradiated by nuclear disintegration products.

The highly efficient detection of gamma rays by the use of organic phosphors (e.g. anthracene) has led to the widespread adoption of the scintillation counter for this purpose.

GENERAL

Vacuum pumps have undergone many changes in design since their introduction in 1915 – particularly the use of oil as a pumping fluid (Burch 1928) – but they have changed little in principle and even today a satisfactory theory of their operation is lacking. Indeed there is little doubt that the provision of a satisfactory theory covering the operation of diffusion pumps forms one of the basic needs of design at the present time. Although it is obvious that the speed of a pump is basically determined by the physical size of the input aperture, it must be admitted that much equipment is underdimensioned because of the ignorance of this fact. Backstreaming and ultimate pressure remain serious problems, particularly in the very high energy electro-magnetic accelerators. There are signs of a return to the use of mercury pumps in the smaller machines, where the cost of providing liquid air for the necessary traps is not excessive. The stability and freedom from contamination problems are highly attractive.

The introduction of rubber 'O'-ring seals has resulted in improved reliability, although, with the thousands of gaskets required, leak hunting is still a major item in the training of any nuclear physicist. It is, in general, unnecessary to calibrate gauges with any degree of accuracy, the successful running of the machine being in itself the best possible indication of satisfactory operating conditions.

Vol. 1 1951

VACUUM-POWDER INSULATION

for low temperatures

L. L. Katan, A.R.C.S., D.I.C., Ph.D.

Research & Development Department,
*British Oxygen Co. Ltd., London, S.W.19**

Summary

A REVIEW is given of the development of vacuum-powder insulation, from the early experiments of Smoluchowski and Dewar to the present day and the importance of thermal insulation at very low temperatures is explained and emphasised. The theory of thermal conduction of porous materials is then briefly considered in terms of the variables involved, with particular reference to pressure. Recent experiments at low temperatures on four typical insulants over a pressure range of 760 mm. to 0.001 mm.Hg are described, and the results correlated theoretically. A brief outline is given of industrial applications of the method, and substantial developments in the future are envisaged.

Sommaire

UN COMPTE rendu du développement de l'isolement par poudre vide depuis les premiers travaux de Smoluchowski et Dewar jusqu'à nos jours. L'importance de l'isolement thermique aux très basses températures est souligné. La théorie de la conductivité thermique de matériel poreux est ensuite traitée succintement en fonction des variables engagées, et en rapport particulièrement avec la pression. Des expériences récentes à basses températures, sur quatre isolants typiques sur une gamme de pression allant de 760 mm. à 0,001 mm. Hg sont décrites et les résultats comparés théoriquement. Un bref aperçu est donné des applications industrielles de la méthode, et des développements d'envergure, futurs, sont envisagés.

INTRODUCTION

UNTIL FAIRLY RECENTLY, all methods of thermal insulation could be classified into two groups. In the first of these some kind of solid material was employed, in contact with the surface to be insulated, no attempt being made to control the pressure within the lagging space. In this context, solid material must be taken to include substances with open structure such as slag wool and cork. In the second group, no solid was used, but the pressure in an air-space around the surface to be insulated was reduced, thus reducing the conduction and convection of the air within the air-space. Usually the surface to be insulated, as well as the surface at ambient temperature, was highly polished to minimise radiation. The application of this method to the Dewar flask is, of course, well known.

* M.S. Received July, 1951.

In this paper a third technique is described, which has certain advantages over both the others. In this method both of the older methods are used simultaneously, *i.e.*, the pressure is reduced in a lagging space filled with an open-structure insulant.

Historical

The first published reference to the method appears to have been by Smoluchowski in 1910[1], and early patents were taken out in 1909 by Fate, and in 1913 by Stanley[2]. With the rapid development of the ordinary Dewar vessel, interest in vacuum-powder insulation diminished, although Dewar himself found that filling the space in a vacuum vessel with certain powders gave an increased insulating efficiency-almost equal to that obtained by silvering the walls.

In 1926, Aberdeen and Laby[3] measured the thermal conductivity of ' Silox ', a very pure form of powdered silica with a bulk density of 0.04 g./c.c. over

a wide pressure range from atmospheric downwards. They found that their results in the pressure range 1-760 mm. Hg could be approximately expressed by :

$$k = \tfrac{1}{2} k_o \log_{10} p/n \qquad (1)$$

where k = conductivity of the powder
k^o = function of the conductivity of the gas in the interstices of the powder
p = pressure (mm. Hg)
n = constant, characteristic of the gas.

They pointed out the possible use of the method for insulating liquid oxygen containers.

Then little appears to have been done until 1933, when Kannaluik and Martin[4] investigated the effect of pressure on the thermal conductivities of powdered magnesium oxide, carborundum, glass, and diphenylamine in hydrogen, air, and carbon dioxide. They confined themselves to the pressure range 1-80 mm. Hg, and their results are consequently of limited application. They showed, however, that Aberdeen and Laby's equation was not strictly true even over a narrow pressure range.

In 1934, Kistler and Caldwell[5] extended the work of Aberdeen and Laby by studying the conductivity of silica aerogel over the pressure range 760 to 0.00007 mm. Hg. Their results confirmed those of Aberdeen and Laby, but they did not feel justified in deriving equation (1).

A good deal of doubt appears to have existed in the early days as to whether the powder functioned as an insulator in itself, or merely assisted the attainment of high vacuum by sorption of residual gas. By 1939, however, it was quite clear that the vacuum-powder method was more efficient than either vacuum or powder alone; and that the improvement could not be solely due to sorption.

The first satisfactory general theoretical analysis was given in 1939 by Austin[6], and the first of the recent patents is held by Aktiebolaget Termisk Isolation, Stockholm, dated 1939[7] and others by Linde Air Products Co., U.S.A.[8]

The most recent published work is by Blatt, Bresler and Ryabinin[9], who confirmed the results of Kistler and Caldwell.

Importance in the Very Low Temperature Field

The method is particularly attractive in the very low temperature field as efficient thermal insulation is of great importance for the following reasons.

To carry out a process at a temperature below ambient it is necessary to transport all the heat which leaks into the system to the higher temperature. This can only be done by the performance of extra work; the amount of this work being given for an ideal case by the second Law of Thermodynamics, *viz.* :

$$\frac{q}{w} = \frac{T_2}{T_1 - T_2}$$

where w is the amount of energy necessary to transport the quantity q of heat (w and q in the same units), from T_2 to a higher temperature T_1. It will be seen that the coefficient of performance of the system, q/w, is diminished by a fall in T_2. For example, with an ambient temperature T_1 of 20°C and an operating temperature T_2 of –20°C, the coefficient of performance is about 6.3, but at liquid oxygen temperature,–183°C, it is only 0.44. Thus the energy requirement to eliminate ' cold loss ' in a liquid oxygen system is about fourteen times that required to eliminate the same loss at –20°C.

Moreover, at the lower temperature, the cold loss through given insulation is itself larger since the conduction of heat is proportional to the temperature difference. If plain vacuum insulation is used, the cold loss is actually dependent on a function involving the fourth power of the temperatures concerned.

Finally, the technical efficiency of refrigeration machines and methods decreases with falling temperature. It is evident therefore that the efficiency of thermal insulation at very low temperatures, *e.g.*, in liquid air installations where the energy requirements for temperature-maintenance may be a hundredfold those at ordinary refrigeration levels, is of vital importance.

Recent Developments

The last ten years or so have seen a marked increase in the use of very low temperature processes. In particular, one may refer to the low temperature separation of hydrocarbons, and to the production,

Vol. 1 1951

storage, and transport, of the liquefied components of air. In the latter connection, for example, if it is noted that the volume of gaseous oxygen is about 800 times that of liquid and that the liquid need not be stored under pressure, it will be evident that handling of oxygen as liquid has substantial advantages over handling as gas. In fact, most of the high purity oxygen used is stored and transported in the liquid form. However, since it is usually impractical to maintain the liquid greatly below its boiling point, any heat inleak leads to a corresponding waste by evaporation. Thus efficient thermal insulation is of paramount importance in the industries dealing with liquefied gases.

It is for this reason, therefore, that attention has been focused on the vacuum-powder method of thermal insulation, since this has promise of being the most efficient insulating method available. Within recent years storage tanks and transfer lines for liquefied gases have been developed both by the Linde Air Products Company in the U.S.A., and the British Oxygen Company in the U.K. A fundamental study of the subject has also been carried out in the Research and Development laboratories of the British Oxygen Company. The present paper sets out, *inter alia*, some of the results of this study.

THEORETICAL

General Theory of Heat Conduction

The conduction of heat through a material macroscopically at rest is governed by Fourier's law, *viz.*,

$$dq'/dt = -k\,A\,dT/dx \qquad (2)$$

where dq'/dt = rate of heat transfer (cal. sec^{-1})
k = thermal conductivity (cal. $sec.^{-1}$ $cm.^{-1}$ $°C^{-1}$)
A = area across which heat flows normally ($cm.^2$)
dT/dx = temperature gradient with respect to distance along heat path ($°C.\ cm.^{-1}$)

Several systems of units are in common use; the one shown in parentheses will be used in this paper.

To integrate equation (2) it is necessary to consider to what extent each variable is a function of the others.

(a) It has been established that the rate of heat transfer dq'/dt is not a function of time, *i.e.*, once a steady conduction state is established, dq'/dt does not change with time. Thus we may replace dq'/dt by q and derive the total heat transfer in a time t, if required, from the product $q\,t$.

(b) Provided the insulant is uniform and that it contains no heat source it is evident that T and x are independent variables.

(c) The area, A, is dependent upon the geometry of the system and thus is necessarily a function of x. A convenient way of dealing with this point is by the use of 'shape-factors' described below.

(d) The thermal conductivity, k, has been shown experimentally not to be dependent upon q,[10] and, provided the insulant is uniform, is evidently not dependent on x. However, k is a property of the insulant used and has a fixed value only for a given insulant in a given physical state. Its dependence upon pressure and other factors will be considered later; for integration of equation (2) it is only necessary to consider the effect of temperature, other variables (*e.g.* pressure and density) being regarded as constant.

The variation of k with T is a characteristic function of the insulant[11] and is usually expressed in the general form,

$$k = k_0(1+\alpha T+\beta T^2+\gamma T^3+\ldots) \qquad (3)$$

where k_0 is the thermal conductivity at a reference temperature ($k=k_0$ when $T=0$) and α, β, γ .... are constants usually determined empirically.

Replacing dq'/dt by q, equation (2) may be written

$$q\frac{dx}{A} = -k\,d\,T$$

and hence, from equation (3)

$$q\frac{dx}{A} = -k_0\,(1+\alpha T+\beta T^2+\gamma T^3+\ldots.)\,dT$$

which can now be integrated to give equation (4)

$$q\int_{x_1}^{x_2}\frac{dx}{A} = k_0(T_1-T_2)\left\{1+\frac{\alpha}{2}(T_1+T_2)+\frac{\beta}{3}(T_1^2+T_1T_2+T_2^2)+\ldots\right\} \qquad (4)$$

where the heat path is from x_1 at T_1 to x_2 at T_2.

Instead of dealing with the cumbersome expression for conductivity on the right hand side of equation (4), it is usual to define the *mean thermal conductivity*, k_m, over the temperature range T_1 to T_2 by–

$$k_m = \frac{\int_{T_1}^{T_2} k\,dt}{T_1 - T_2}$$

$$= k_0\left\{1 + \frac{\alpha}{2}(T_1+T_2) + \frac{\beta}{3}(T_1^2+T_1T_2+T_2^2) + \ldots\right\}$$

and hence equation (4) becomes

$$q\int_{x_1}^{x_2} dx/A = k_m\,(T_1-T_2) = k_m\,\Delta T \qquad (4a)$$

where $\Delta T = T_1 - T_2$

The value of $\int_{x_1}^{x_2} dx/A$ depends on the geometrical relationship between A and x, and it is only in a few simple cases that the integral can be exactly evaluated. For practical purposes it is convenient to *define* a 'shape-factor', S, by

$$\frac{1}{S} = \int_{x_1}^{x_2} \frac{dx}{A}$$

so that equation (4a) becomes

$$q = k_m\,S\,\Delta T \qquad (5)$$

Shape factors. Detailed discussion of shape factors is outside the scope of this paper. For certain simple geometrical configurations, the shape factor can be determined exactly, *viz.* :

(a) For concentric spheres, radii r_1 and r_2, areas A_1 and A_2,

$$S = \frac{4\pi r_1 r_2}{r_2 - r_1} = \frac{\sqrt{A_1A_2}}{r_2 - r_1}$$

(b) For large parallel plates (*i.e.*, when edge losses can be ignored), areas A, distant x apart,

$$S = A/x$$

(c) For long cylinders (*i.e.*, when end losses can be ignored), length L, radii r_1 and r_2, areas A_1 and A_2

$$S = \frac{2\pi L}{\log_e r_2/r_1} = \frac{A_2 - A_1}{(r_2 - r_1)\log_e A_2/A_1}$$

Approximate values can be found in other cases, sometimes algebraically and sometimes graphically.

Thermal conductivity. The thermal conductivity of all substances is dependent on temperature, as already shown by equation (3). The values of the constants α, β, γ, etc., are variable from substance to substance, not only in magnitude but also in sign; usually only α and β are of importance, the further terms being negligible. For most metals α is negative, *i.e.*, conductivity decreases with temperature; for many non-metals the reverse is true. In particular, the conductivity of air increases with temperature.

In general, however, temperature effects are small, and rarely exceed 0.2% per °C, *i.e.*, the conductivity at liquid air temperature differs by 40% from that at room temperature for the most temperature-variable materials. (At temperatures approaching the absolute zero, *e.g.*, below 10°K, remarkable changes in thermal conductivities may indeed occur. Consideration of these is outside the scope of this paper).

For most practical purposes, use of the mean value for conductivity over the temperature range of interest eliminates the necessity of making detailed investigation of temperature effects.

Thermal conductivities at normal pressures vary over a very wide range. At one extreme are metals such as copper and silver, with conductivities around 1 cal. sec.$^{-1}$ cm.$^{-1}$ °C^{-1}; at the other, are gases with conductivities in the range 5 to 10 x 10^{-5} cal. sec.$^{-1}$ cm.$^{-1}$ °C^{-1}. The best insulants are within this latter range, and attention will now be confined to these.

Radiation and Convection

The general theory of porous insulation is dealt with below from the point of view of conduction; but it is also necessary to consider the effects of radiation and convection.

In a space containing a gas at atmospheric pressure, *e.g.*, air, most of heat transfer is usually by convection but at large temperature differences radiation is also

Vol. 1 1951

important. Both of these mechanisms of heat transfer are virtually eliminated by filling the space with a porous material, although with materials of exceptionally low bulk densities (*i.e.* high porosities) or open structures, some convection may occur.

Effect of Pressure on Thermal Conductivity of Porous Insulants

The general effects of pressure changes on the conductivity of gases are well known, and can be explained rigorously on the basis of the kinetic theory. For porous insulants the effects are now fairly well known but the theoretical explanation of them, *e.g.* equation (1), has not been complete. The following is a theoretical explanation of the effects of pressure on the thermal conductivities of gases and porous insulants, in the light of the most recent evidence. The effects are illustrated in Fig. 1 with air as the gas concerned. Although the argument is general, numerical values apply to air.

In a gas-filled vessel, the conductivity remains constant as the pressure is reduced from atmospheric, until a pressure is reached at which the mean free path of the molecules approaches the distance between the hot and cold walls of the vessel. The conductivity then decreases, being directly proportional to the pressure. For vessels of moderate size, *i.e.*, when the inter-wall distance is from 5 to 50 cm., the transitional pressure at which the conductivity starts to fall off is 10^{-3} to 10^{-4} mm. Hg and the pressure at which the conductivity is 10% of its normal value is hence 10^{-4} to 10^{-5} mm. Hg. Such pressures are attained in Dewar vessels. At zero pressure the conductivity is also zero.

Porous insulants show an analogous behaviour, with the important difference that the transitional pressure is that at which the mean free path of the gas molecules becomes of the same order of length as the interstitial distance, *i.e.*, the mean distance between particles. It is thus independent of the size of the vessel, and is very much higher than for a pure gas. For example, with a coarse material such as slag wool, the interstitial distance is of the order of 1 mm., consequently the transitional pressure is about 0.1 mm. Hg and the pressure at which the conductivity is 10% of its normal value should be 0.01 mm. Hg. With a fine powder such as basic magnesium carbonate the interstitial distance is nearer 0.1 mm., so that the transitional pressure is about 1 mm. Hg and the conductivity reaches 10% of its normal value at 0.1 mm. Hg.

With Santocel, a form of silica aerogel, the interstitial distance of which is extremely small, the transitional pressure exceeds atmospheric, *i.e.*, at atmospheric pressure the conductivity is less than that of still air.

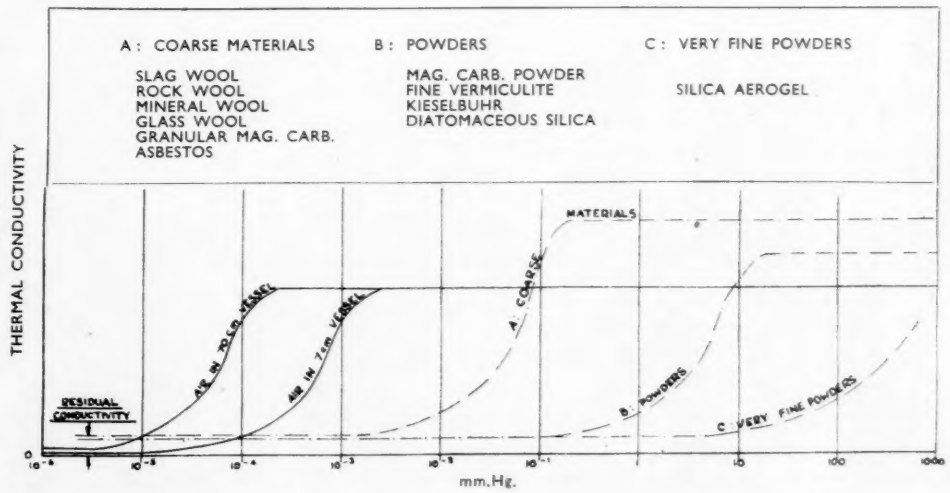


Fig. 1. Idealised representation of the effect of pressure on thermal conductivity.

With porous insulants, as the pressure is reduced below the transitional the conductivity decreases as with pure gases, but not to zero, since there is a 'residual' conductivity due to direct conduction through contact between solid surfaces. This residual conductivity, which is negligibly affected by pressure, is very small for dry, powdered or fibrous materials, and may be neglected for practical purposes.

EXPERIMENTAL

General Procedure

All the experimental work to be described was carried out at liquid air temperatures, since it is chiefly for these that vacuum-powder insulation is of special interest. The results, however, are not specific to the temperature range dealt with and could be applied, with quantitative modifications, to any temperature.

The chief difficulty in investigations of the conductivity of porous materials at low pressures, is that the conductivities are so low as to make end-effects very serious. For example, if the Lees disc method were used, the conductivity of the air surrounding the test block, and of the container of the insulant would exceed that of the sample under test. The difficulty has been overcome by testing a *continuous* layer of insulation, located around a suitable vessel. This (inner) vessel was cooled with liquid oxygen or nitrogen, the evaporation rate of which gave an accurate measure of the rate of heat transfer. For work at higher temperatures solid carbon dioxide would be convenient, and for temperatures exceeding atmospheric the inner vessel could be heated electrically. This last method was used by Garner, Suchell and Adams[12], following on the work of Groeber[13], although as they were also interested in low temperatures, they surrounded their outer vessel with liquid air and employed the heater merely to maintain a normal temperature. In the present work, the outer vessel was maintained at atmospheric temperature so as to give values for k_m over the temperature range of practical interest.

Several vessels have been employed, and two typical ones are illustrated in Fig. 2. The complete experimental arrangement, including vacuum and metering systems, is shown in Fig 3. The procedure was as follows.

The vessel was charged with liquid oxygen, and allowed to reach thermal equilibrium. The rate of heat transfer was then determined by measuring the rate of evaporation of the liquid by means of a gas meter. Thermal equilibrium was established by leaving the system for 12 to 15 hours, during which the inner vessel was kept never less than 80% full of liquid by topping up from time to time. Readings of evaporation rate were always taken to the stage at which 50% of the liquid had evaporated, and in many cases until all had gone.

Temperatures of the outer vessel were measured either by thermocouples soldered to the wall, or by thermometers encased in aluminium foil (in intimate contact with the wall) and covered by wax.

Analysis of experimental results. To illustrate the general nature of the results obtained, those for a typical experiment are given below. Vaporisation rates for this experiment are shown in Fig. 4.

Experiment C2-9. Apparatus consisted of coaxial cylinders (Fig. 2a) inner diameter of insulation 15 cm., outer 35 cm.; inner height 32 cm., outer 40 cm. Insulation tested was 'Stillite' brand of slag wool nodules, at a bulk density of 13.8 lb/cu.ft. Ambient : 23°C. Barometer: 762 mm. Hg. Pressure in insulation : 4.5 mm. Hg.

Temperature measurements				MEAN	
				Individual	Total
Volume of gas evolved (litres)	300	840	2000		
Temperature 2 in. from top of wall (°C) ...	19.0	19.8	19.0	19.3	19.0
Temperature 2 in. from bottom of wall (°C) ..	18.0	18.8	18.9	18.6	

The graph of vaporisation rate (dv/dt) against total volume evaporated (or liquid level) shown in Fig. 4 was extrapolated back to the point at which the vessel was full. The extrapolation was made from the smooth and moderately level part of the curve, since it was considered that the variations in rate

Vol. 1 1951

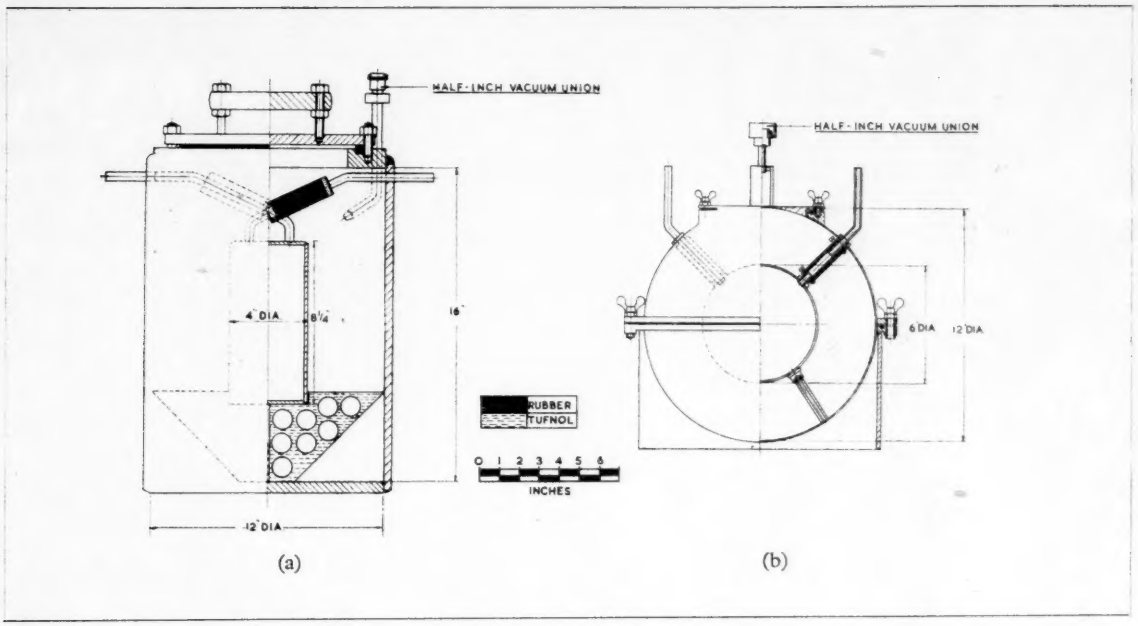


Fig. 2. Detail of thermal conductivity test apparatus. (*a*) Cylindrical, (*b*) Spherical.

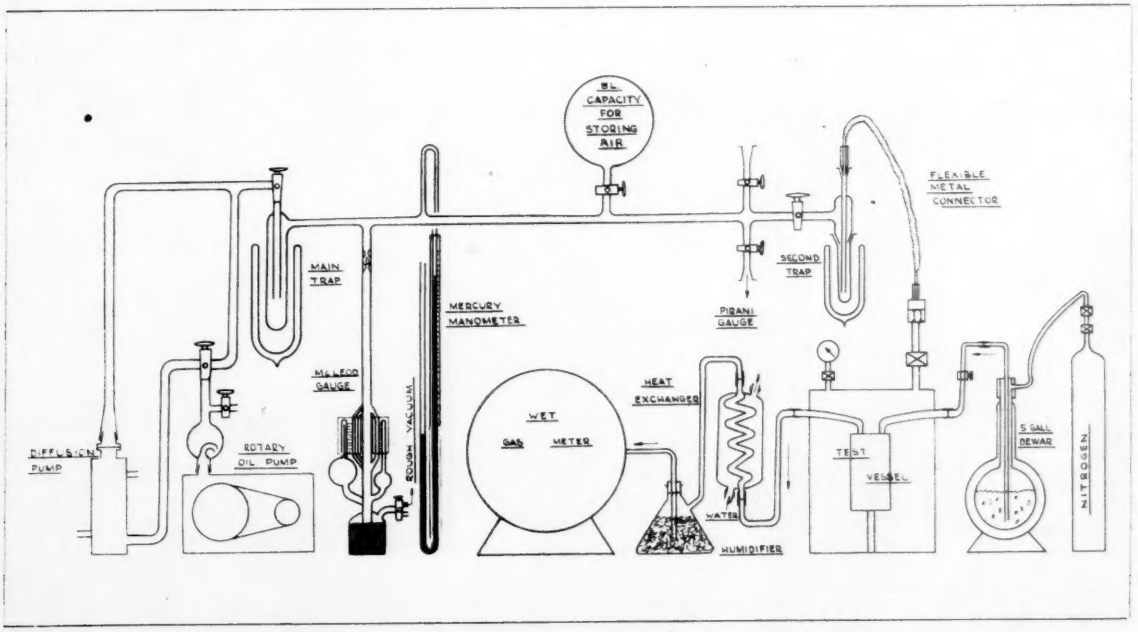


Fig. 3. Experimental set-up.

encountered in the early stages of the experiment were due to unsteady conditions (in particular, to the splashing of liquid droplets into the outer tube, and to super-heating of the liquid oxygen).

From the extrapolated value for dv/dt was obtained q, the total rate of heat transfer (1 litre/min. evaporated is equivalent to 1.215 cal/sec.) and from this, k_m, the mean thermal conductivity, by the methods already described. The shape-factor for this vessel was 297 cm.

Pressure Range

Experiments were carried out with pressures in the insulation space from atmospheric down to about 0.001 mm. Hg. In some cases the pressure range was limited by the vapour pressure of the material used.

Materials

Four materials were chosen as representative of the types available, and also as being of commercial interest. They were as follows :

(*a*) *Slag wool.* This is a non-combustible fibrous material, made by blowing high pressure steam into molten furnace slag, which is converted into a mass of entangled fibres. ' Stillite ' brand was used, this containing less ' shot ' and being more convenient to handle than most varieties. The packing density was varied, most of the experiments being carried out with material of 16 lb/cu. ft.

(*b*) *B.M.C. powder.* This is precipitated basic magnesium carbonate of particle diameters mostly within the range 0.05-0.08 mm., consisting of isotropic non-crystalline matrices containing minute crystals. Its formula is approximately $3MgCO_3.Mg(OH)_2.4H_2O$. The bulk density used was 8 lb/cu. ft.

(*c*) *Brelite.* This is an expanded mineral of volcanic origin, supplied by Vermiculite Ltd. It consists of spherical aggregates of cellular structure, and has the remarkably low bulk density of 6 lb/cu. ft. It has only very recently become available.

(*d*) *Silica aerogel.* This is made by dehydrating silica gel in such a manner as to avoid collapse of the cell structure. It is supplied under the name of ' Santocel ' by Monsanto Chemical Co. in the form of a very fine powder with a bulk density of 5 lb/cu. ft. The size of the extremely fine pores in the material is stated to be of the order of 10^{-5} mm. [5,9]

RESULTS AND DISCUSSION

The results obtained for thermal conductivities of the four typical insulants are shown in Fig. 5 plotted against pressure. Results obtained by earlier workers for silica aerogel agree well with the present results for Santocel although identical agreement is not to be expected in view of possible differences between the grades of material used.

The results qualitatively support the theory already given, although exact correlation is difficult since the materials used were not uniform. However,

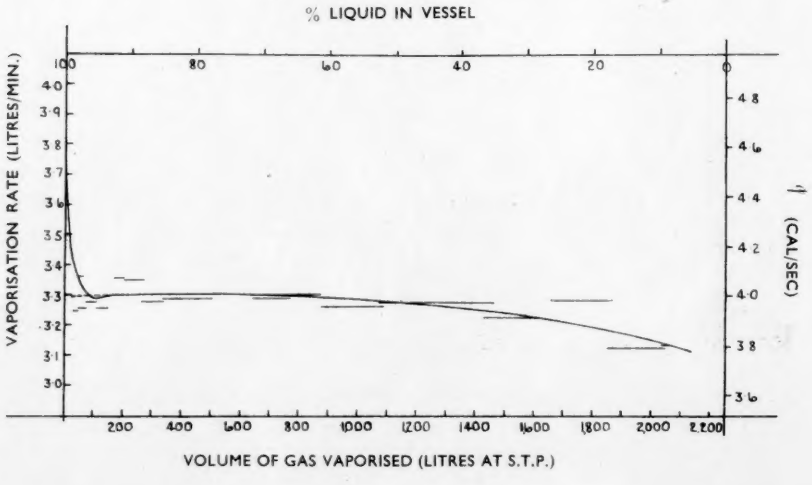


Fig. 4. Expt. C2-9. Vaporisation rate *vs* liquid level.

Vol. 1 1951

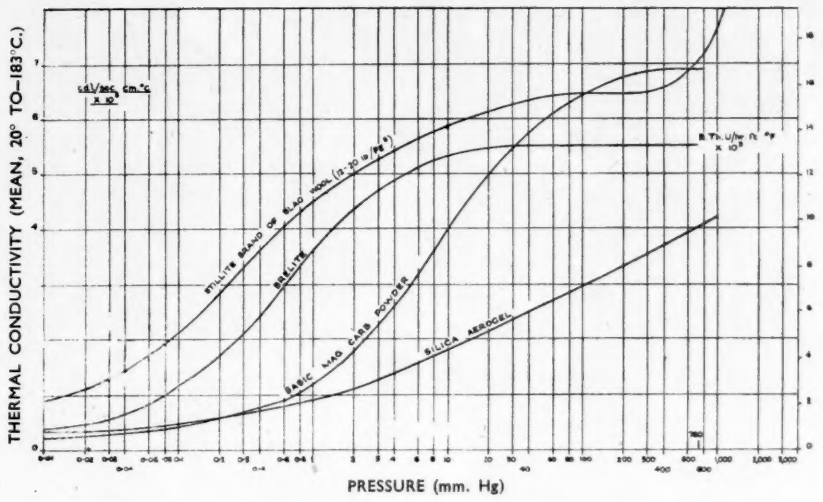


Fig. 5. Effect of pressure on thermal conductivity of insulants.

if a reasonable estimate be made from Fig. 5 of the pressures at which the constant conductivity region characteristic of atmospheric pressure changes to the region where conductivity is directly proportional to pressure, the following values ensue :

	Transitional Pressure (Hg)	Corresponding Mean Free Path at 20°C.
Slag Wool	0.3 mm.	0.17 mm.
Brelite	1 mm.	0.05 mm.
B.M.C. Powder	10 mm.	0.005 mm.
Silica Aerogel	>760 mm.	$<10^{-4}$ mm.

The value for the mean free path corresponding to the transitional pressure agrees well, in each case, with the mean interstitial distance.

The improvement in thermal insulation effected by pressure reduction is illustrated in Table 1, where thermal conductivity is given as a proportion of its normal value.

It will be seen from Table 1 that the thermal conductivity of insulants is markedly reduced at quite moderate vacua. Thus to reduce the thermal conductivity of B.M.C. powder to 10% of its normal value, it is only necessary to evacuate to about 0.1 mm. Hg. To reach the equivalent of such conductivity in a Dewar vessel would require evacuation to below 0.0001 mm. Hg which would require considerably more equipment and time. These effects are also illustrated in Fig. 6.

TABLE 1.—THERMAL CONDUCTIVITIES AT REDUCED PRESSURES

Pressure (mm.Hg)	Thermal Conductivity as % of Normal Value			
	Slag Wool	Brelite	B.M.C. Powder	Silica Aerogel
760	100	100	100	100
100	91	100	94	70
10	83	97	58	43
1	63	65	17	21
0.1	30	21	7	11
0.01	12	7	3	8

APPLICATIONS

General

The actual choice of the method of insulating a vessel at low temperature is dictated by economic considerations, *i.e.*, by the relative costs of cold

production and insulation. It can, however, be asserted generally that the cost of refrigeration at liquid air temperature is so large as to make any method of insulation which is superior to conventional methods worthy of consideration.

As has been already stated, the thermal conductivity of the best conventional insulants is similar to that of still air. Improvement can be effected either by reducing the pressure or the interstitial distance in the lagging : in both cases the fundamental change is to reduce the frequency of molecular collisions in the gas phase. Reduction of the interstitial distance below the mean free path of air molecules is difficult, and silica aerogel is the only readily available material in which it has been effected. The conductivity of Santocel brand of silica aerogel is about 60% that of still air.

Thus pressure reduction is the only method which will greatly reduce thermal conductivity, and as described already, it is very much more effective if used in conjunction with a porous insulant. From the industrial point of view this means that vessels can be constructed in which very much larger leakage rates can be tolerated than are permissible in Dewar vessels. To illustrate this point, consider two vessels of the same capacity, the first insulated by vacuum alone, and the second by vacuum powder. For comparable insulating efficiencies, the insulation space for vacuum powder will be about three times that for plain vacuum. If the latter volume is 100 litres, and it is considered permissible to re-evacuate twice a year, the maximum tolerable leakage rate will be about 10^{-7} lusec, taking 10^{-5} mm. Hg as the maximum permissible pressure. For the vacuum-powder insulated space of 300 litres, taking the maximum permissible pressure rise as 0.05 to 0.1 mm. Hg the maximum tolerable leakage rate will be about 10^{-3} lusec. Whereas 10^{-7} lusec would be virtually impossible to attain in a vessel of any size even using charcoal sorption or getters, 10^{-3} lusec is not beyond the reach of modern vacuum engineering.

Storage Tanks

Heat transfer is roughly proportional to surface area whereas the capacity of a tank is proportional to its volume, consequently the ratio of the two is inversely proportional to a linear dimension, *i.e.*, cold losses decrease in importance with increase in size.

Vacuum-powder is therefore most valuable for tanks of small or moderate size, although the method has been employed up to a size of 1200 cu. ft. (equivalent to 1 million cu. ft. gaseous oxygen capacity). For the very largest tanks, losses with normal insulation are so small that the complication of vacuum insulation becomes unnecessary.

The problems associated with the construction of these tanks are numerous, the more important being as follows.

(*a*) Evacuation of a powder-filled volume is a slow process because of the resistance of the powder, and also, in many cases, because of the evolution of vapour from the powder. Examples can be quoted where several days were required to evacuate a volume of a few cu. ft. to 0.1 mm. Hg in the absence of leaks.

(*b*) Several connections to the storage vessel are essential, and these must necessarily pass through the vacuum space. At the same time they should not conduct too much heat. This problem is twofold, therefore, firstly to construct joints vacuum-tight both at normal and at liquid air temperature, secondly to provide some kind of heat break.

(*c*) With a temperature drop of about 200°C in the storage vessel contraction stresses are serious, and must be carefully taken into account.

(*d*) Choice of materials for use down to -200°C is limited. Copper stainless steel and aluminium are satisfactory, but mild steel is too brittle to be used. Rubber and other materials plastic at normal temperatures can only be used if special precautions against fracture are taken.

Transport Tanks

Losses in transport of liquefied gases due to evaporation are always serious, but are of exceptional importance when the material has to be carried a great distance. Very little has been published on the subject, although there is an account of a rail-car which has a capacity of 900 cu. ft. (equivalent to

Vol. 1 1951

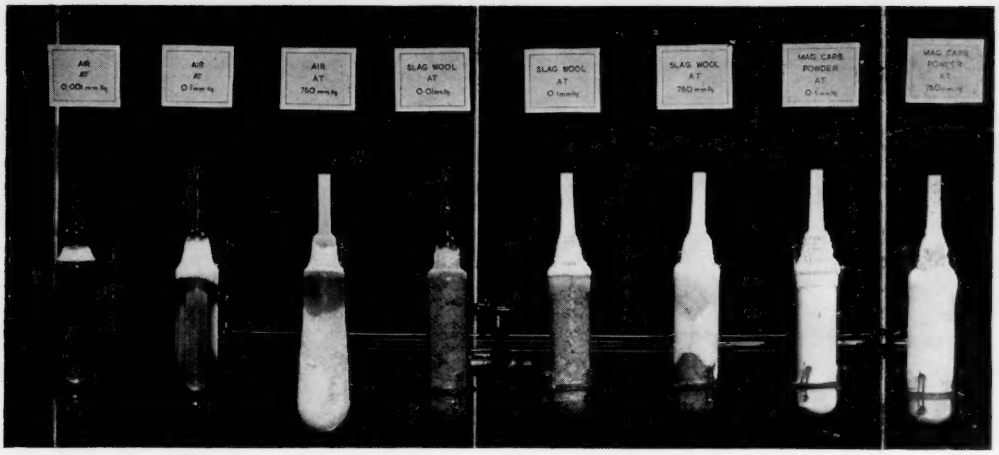


Fig. 6. Each tube contains boiling liquid oxygen and is surrounded by an insulation space of fixed dimensions. These are such that the condition for absence of dewing is a reduction of thermal conductivity to 10% of the normal (atmospheric pressure) value. It will be seen this reduction is obtained with B.M.C. powder at 0.1 mm. Hg, slag wool at 0.01 mm. Hg, or air at 0.001 mm. Hg. At higher pressures, insulation efficiency is less and dewing or ice-formation occurs. On all the tubes at atmospheric pressure ice-formation is pronounced, but the one without insulation is much the worst. The shape of the ice-crust indicates that convection is an important factor in this case. The ice-crust around the upper part of each tube is due to heat transfer by conduction and radiation through the glass.

750,000 cu. ft. free oxygen). This, of course, is only appropriate for very long journeys.

The problems associated with the development of transport tanks are similar to those encountered with storage tanks, although conditions of wear and tear are more severe.

Transfer Tubes

A very recent development is the use of vacuum powder insulation in transfer tubes for liquefied gases. Such tubes are employed extensively in liquid air plants, and the introduction of vacuum-powder insulation can effect substantial economies. Instead of utilising all the increased efficiency of vacuum-powder insulation for reduced heat transfer, some may be applied to reducing the thickness of insulation. The saving here is substantial, since insulating efficiency of tubes follows an exponential law with regard to thickness. For example, when insulating a 1 in. diameter tube, the same insulating efficiency would be obtained from $\frac{1}{2}$ in. of B.M.C. powder at 0.1 mm. Hg as from 42 ft. of the same material at atmospheric pressure. This reduction in the quantity of insulant used, is of particular importance for plant used intermittently, since the amount of energy required to heat or cool the insulation to equilibrium may be a substantial proportion of the total consumption.

Vacuum-powder insulated tubes may also find applications in the high temperature field, *e.g.*, steam transmission, and although for many applications it is unlikely that the extra cost would be justified, the method may prove of great value where special problems are encountered, such as space-limitation or a need for maximum efficiency.

CONCLUSIONS

Enough examples have been given to show that the subject of vacuum-powder insulation is one that is actively developing at the present time. The theoretical basis seems to be fairly well established, and follows the lines given in this paper; practical applications, however, although increasing rapidly, are still limited.

The problems involved are largely those normal in

vacuum technology coupled with engineering problems peculiar to thermal insulation. Low temperature work presents some extra difficulties. There is no doubt, however, that the next ten years or so will see great advances made, perhaps at high, medium, and normal refrigeration temperatures; certainly at very low temperatures.

The writer is glad to acknowledge his debt to colleagues of the Research & Development Department of the British Oxygen Company, whose work he has freely quoted; in particular to Mr. B. A. Curry, Dr. J. B. Gardner, Messrs. A. Monroe and N. W. Rōberts.

The writer's own work was carried out under the supervision of Mr. A. M. Clark, Research Manager Gas Separation, whose help and advice are also gratefully acknowledged.

This paper is published by permission of the directors of the British Oxygen Company Ltd.

References

[1] SMOLUCHOWSKI, *Bull. In. Acad. Cracovic*, 1910, 129.
[2] FATE, Brit. Patent 12022/09
STANLEY, Brit. Patent 17832/13.
[3] ABERDEEN and LABY, *Proc. Roy. Soc.*, A., 1926, **113**, 459.
[4] KANNALUIK and MARTIN, *Proc. Roy. Soc.*, A., 1933, **141**, 144.
[5] KISTLER and CALDWELL, *Ind. Eng. Chem.*, 1934, **26**, 658.
[6] AUSTIN, *Symposium on Thermal Insulating Materials*. (Am. Soc. Testing Materials, Philadelphia Pa, 1939) 49.
[7] AKTIEBOLAGET TERMISK ISOLATION, STOCKHOLM, U.S Patent 2,164,143, (1939).
[8] LINDE AIR PRODUCTS CO., Brit. Patent, 544, 342 (1942).
[9] BLATT, BRESLER and RYABININ, *J. Tech. Phys. U.S.S.R.*, 1945, **15**, 916.
[10] *Loc. cit.* (6) 55.
[11] See for example, NICHOLLS, *Bull. Am. Ceramic Soc.*, 1936, **15**, 37; SEEMANN, *Phys. Rev.*, 1928, **31**, 119.
[12] GARNER, SUCHELL and ADAMS, *Refrig. Eng.*, 1944, **48**, 209.
[13] GROEBER, *Z. für die gesamte Kälte Industrie*, 1909, **16**, 84.

Vol. 1 1951

LETTERS TO THE EDITOR

Heating in Vacuo by an External Radiation Source

Sommaire

UN PETIT OBJET dans un récipient évacué devait être chauffé. Dans ce cas, le chauffage habituel par courent d'Eddy ne pouvant être utilisé un essai fut fait de chauffer par rayonnement. Une lampe à filament de tungstène de 750 W fut placée à l'un des foyers d'un réflecteur ellipsoidal et l'objet à chauffer placé à l'autre foyer. L'expérience réussit. L'objet, dans le récipient évacué était chauffé à 1,300°K. Diverses applications du dispositif sont suggérées, mais il y en a probablement d'autres.

IN THE COURSE of experimental work on vacuum tubes it became necessary to heat metallic and non-metallic electrodes *in vacuo* by means other than the conventional eddy-current heating device ; this was partly due to the shape and material of the electrodes and partly to the bulk of the eddy-current heater which made it impossible to use it in the restricted space available for the experiment. This caused us to enquire whether one could not use the radiation from a tungsten filament lamp.

The temperature of the filament in a gas-filled tungsten filament lamp is normally in the region of 3,000°K ; the equivalent black-body temperature may be anywhere between 2,200°K and 3,000°K. If all the radiation from such a filament were focused into an image the same size as the filament by means of an ideal optical system, an isolated body smaller than the image, at the position of the image, would attain the same temperature as the tungsten filament. With any real optical system the temperature at the image will be lower. It is of interest to estimate the temperatures that can be expected in practice.

Let us consider an ellipsoidal reflector for the optical system. In a complete ellipsoid, (i.e. a surface of revolution about an axis passing through the two foci) all the radiation originating at one focus will converge at the other conjugate focus. For practical reasons it is not feasible to use a complete ellipsoid ; thus we can only use a fraction F of the radiation equal to the solid angle subtended by the ellipsoid at the focus divided by 4π.

Next, we have to take into account that the filament is of finite extent. Thus, its image is larger than its physical dimensions. The actual linear enlargement is estimated to be of the order of

$$\frac{a + f}{a - f}$$

where a is the longer half-axis and f the distance of the focus from the centre of the ellipsoid. This relation only holds as long as the object is very small compared with the ellipsoid.

Finally, we have to consider losses of radiation by absorption, reflection and scattering at the reflecting surface and by the glass walls containing the source of radiation, and perhaps surrounding the body to be irradiated. These losses are difficult to compute in general, and will be taken into account by multiplying the energy flux going into the image with a factor E (smaller than unity).

Provided the body, which is to be irradiated, is a thermally insulated sphere and loses heat only by radiation, we can calculate T_2, the temperature attained by the sphere by equating the energy received with the energy radiated by the sphere. We make moreover the assumption that the sphere is equal in size to the image of the source of radiation (also of spherical form). This leads to

$$T_2 = T_1 \sqrt[4]{F.E\left(\frac{a - f}{a + f}\right)^2}$$

where T_1 is the equivalent blackbody temperature of the filament. When reasonable values for the parameters are inserted into this equation it is found that temperatures between 1,000°K and 1,500°K should be obtainable.

A number of experiments have been carried out with ellipsoidal reflectors of various sizes and shapes, and of various surfaces, in conjunction with a variety of lamps with tungsten filaments. The relation given above is roughly confirmed; an ellipsoidal reflector having the following dimensions :

$$\begin{aligned} a &= 6.5 \text{ inches} \\ f &= 2.5 \text{ inches} \\ F &= 0.7 \end{aligned}$$

produced a temperature of about 1,300°K on a body (actually the anode of a valve) *in vacuo*. A thin nickel-iron alloy sheet can be heated to nearly the same temperature in air.

With a 750-watt tungsten filament lamp it is possible

to heat objects of nearly 3 cm^2 surface area to bright red heat, the exact temperature depending largely on the shape and surface texture of the object.

The concentrated heat source consisting of a tungsten filament lamp in combination with an ellipsoidal reflector has been used for a number of purposes in this laboratory :

(1) Outgassing small electrodes in vacuum tubes.

(2) Firing getters in vacuum tubes.

(3) Heating of indirectly heated cathodes where the magnetic field of the usual heater filament was objectionable.

(4) Localised heating in air or inert atmospheres where the use of an open flame is deleterious or impossible.

(5) Soldering and brazing of small metal parts.

Other uses will undoubtedly be found as the occasion arises and as the knowledge that such a source of heat is available spreads.

A. Leemans.
R. Kompfner.

Clarendon Laboratory,
Parks Road,
Oxford.

15th June, 1951.

Evaporation of Silica

Sommaire

L'AUTEUR REND compte des méthodes employées dans son laboratoire pour la production de dépôts de quartz, ayant de bonnes propriétés d'adhésion, par le procédé dévaporation. Il a été trouvé qu'il est virtuellement impossible d'évaporer du quartz sans une réduction, du moins partielle, en SiO. Il a aussi été trouvé que tous les matériaux réfractaires usuellement utilisés comme creusets sont oxydés en SiO, cette réduction résultant en une contamination du dépôt. Cependant si l'on prend les précautions nécessaires, ceci n'affecte pas l'adhérence du film sur la base. Quelques détails des méthodes employées dans ce sens par le laboratoire sont indiqués. Un compte-rendu plus détaillé de ce travail sera publié ultérieurement.

IN THE FIRST ISSUE of your Review, L. Holland states[1] that he has evaporated silica and produced adhering deposits, although similar experiments conducted by G. Hass and others only yielded flaky deposits. L. Holland placed fused silica into a tungsten spiral, and heated the latter until slow evaporation started.[2]

As the evaporation of silicon and its oxides forms the subject of many a seemingly controversial statement, I would like to summarise some of the conclusions reached in this laboratory. They seem to indicate that :—(a) it is virtually impossible to evaporate SiO_2 without at least partial reduction to SiO, although the SiO vapour and condensed film will under certain circumstances recombine with residual oxygen to yield silica ; (b) all usual refractory metals used for filaments or boats are oxidised during this reduction process, and their oxides are more or less violently evaporated at the same time, so that a corresponding contamination of the deposited silicon oxide film results ; (c) some of these contaminations will markedly decrease the otherwise excellent adhesion of silicon oxides (silicon monoxide and silica) on a clean glass, or freshly aluminised or chromised glass substrate.

It is known that many elements and inorganic compounds attack silica when heated *in vacuo*. Attack by aluminium and magnesium will set in at temperatures as low at 700-800°C, while reduction by carbon only starts at approximately 1,050°C.[3] If silica is heated in tungsten, tantalum or molybdenum spirals or boats at temperatures in excess of say 1,300-1,400°C, it will be gradually reduced to silicon monoxide whose vapour pressure at these temperatures is considerably higher than that of silica, so that it will evaporate much faster than the latter. The liberated oxygen will in turn oxidise the filament metal ; as tungsten, tantalum and molybdenum oxides have vapour pressures much in excess of their parent metal, the metal oxide will evaporate and contaminate the deposited silicon oxide layer. Contamination of evaporated hard silica films by brown tungsten oxide has been reported by J. Yarwood who placed a silica rod inside a tungsten spiral.[4] The same author states that silica placed inside a tantalum cup heated by electron bombardment will evaporate to pure silica films—a conclusion, he has apparently come to by failing to trace colourless transparent tantalum pentoxide contaminations in his silica films. I have found that silica films evaporated from tantalum boats yield a film of higher refractive index than pure silica films, thus indicating the presence of tantalum pentoxide, even when these evaporated films were subsequently heat-treated to transform all

Vol. 1 1951

evaporated silicon and silicon monoxide into silica. Another indirect proof of contamination by tantalum pentoxide seems to be furnished by the gradual oxidation of a tantalum boat by silica ; a very thin tantalum boat, heated to 1,600-1,700°C, is quickly destroyed by silica, and I could find no conclusive evidence of this destruction being caused by a reduction to silicon, and destructive alloying that is known to happen whenever silicon is melted in tantalum.

I have evaporated silicon monoxide from molybdenum and tantalum boats and silica from tantalum and tungsten filaments (or boats) on to clean glass and freshly vacuum-metallised (aluminised and chromised) glass substrates. After the usual thermal treatment, all films exhibited practically the same abrasion resistance, indicating that contaminations by tungsten and tantalum oxides do not decrease the excellent adhesion of silica films.

Quite inferior deposits were on the other hand obtained when evaporating silica from molybdenum boats. Even when films thus obtained seemed perfectly right after their removal from vacuum, they peeled off on exposure to air, and this process was accelerated when immersing them into water. I believe this to be caused by one or both of the following reasons :—(a) contamination by a water soluble molybdenum oxide, such as molybdenum blue or molybdite, formed and evaporated during the reduction of silica to silicon monoxide ; (b) sudden burst of one of these molybdenum oxides, violently evaporated as soon as silica is reduced to silicon monoxide at temperatures probably in excess of 1,300°C. At these temperatures, the vapour pressure of molybdite[5] must be nearly atmospheric, and it might well be that flaky interlayers formed by too rapid an evaporation of molybdite or another molybdenum oxide cause the evaporated silicon oxide to peel off the substrate.

I have deposited colourless and strongly adherent silicon oxide films on various substrates, using a dense and vacuum-preheated cup-shaped carbon crucible with pure silica placed inside. On heating the crucible to approximately 1,800°C, the silica melted and drew into a globule fastly moving in a circle inside the crucible. Stopping the process before the globule fully evaporated, no traces of silicon carbide could be observed either in the crucible or in the evaporated film, but the globule showed pale blue veins believed to be silicon monoxide. Presumably the excess oxygen reacts with carbon to form carbon monoxide that is pumped off by the diffusion pump.[6]

Another of the many possible solutions to deposit pure silicon oxide films consists in slowly evaporating silicon from a beryllia crucible at pressures of 2 to 4 $\times$ 10^{-4}mm. Hg using oxygen as residual gas. Care must be taken to increase oxygen admission when evaporation begins, on account of the powerful gettering action of the silicon vapour and the condensed film. Best results were obtained when a heated glass substrate was placed at a distance from the source approximating the mean free path at these pressures.

A very convenient and relatively low temperature method to evaporate silicon oxide and a number of other oxides or a mixture of oxides with varying refractive indices consists in pelleting a suitable mixture of finely divided oxide and metallic powders, in order to:—(a) reduce a higher oxide to a more volatile lower oxide, without fast evaporation of the reducing metal or its oxide (this can be done if a mixture of silica and beryllium, or silica and chrome powders is used); (b) oxidise a low vapour pressure metal to a more volatile oxide, e.g. silicon to silicon monoxide by oxygen released from chromic oxide or titania ; (c) deposit a mixed oxide e.g. consisting of silica and tungsten powders. Most of these deposits adhere very well, are easily converted into stable high oxides, and have the great advantage over the straightforward evaporation method of high oxides that very little untrapped oxygen is released during evaporation. Thus the metal of the crucible will show little tendency of oxidising and cheap molybdenum heaters will do in most cases. It is planned to publish a full report on similar experiments conducted in this laboratory with a number of electrically and optically interesting metallic oxides.

A. de Rudnay.

Electro Vacuum,
Milserstrasse,
Solbad Hall,
Tyrol, Austria.

1st July, 1951.

[1] HOLLAND, L., *Vacuum*, **1**, (Jan. 1950), 30.
[2] Private Communication from L. Holland.
[3] BERLINER QUARZ-SCHMELZE, Special Catalogue VLK 3507, (Berlin, 1935).
[4] YARWOOD, J., *High Vacuum Technique*, 2nd Edition (Chapman & Hall Ltd., London, 1948), 89.
[5] KELLEY, K. K., *Bull.* 383, Part III, (U.S. Dept. of the Interior, Bureau of Mines, Washington, 1935).
[6] HASS, G. and SCOTT, N. W., *Le Journal De Physique et le Radium*, **2**, (June 1950), 400.

BOOK REVIEWS

La Technique du Vide. (Vacuum Engineering). By MAURICE LEBLANC. (Librairie Armand Colin, Paris, 1951). Pp. 187. Price 4s. 1d.

THE HANDY VOLUMES of the Collection Armand Colin with their familiar blue and white paper covers are well known and popular in France. They are somewhat similar to our own small Methuen monographs, though perhaps less advanced and thus directed to a wider public. Their object is to give concise and simple introductions to a variety of topics in the field of science, philosophy, economics, etc., and those on scientific subjects are of great help to students, to scientists and even to the interested layman. The Physics section comprises quite a few excellent little treatises (*e.g.* Ch. Fabry's books on Thermodynamics, on Electricity, on Heat Conduction, Weiss and Foex's Magnetism and E. Bloch's Kinetic Gas Theory, to mention only a few) and it was with hopeful expectation that the publication of this book on vacuum technique was noted.

The author who did some pioneer work on the use of steam ejectors as vacuum pumps and who, in his own words, had long personal experience in vacuum technique prefaces his book with some remarks about his aims. He explains that the applications of reduced pressures to industrial processes can be divided into two categories. On the one hand we have the use of rough vacuum in many processes of chemical engineering such as fractional distillation, refrigeration, etc.; on the other hand we have the use of high vacuum in processes of a more physical nature such as the manufacture of incandescent lamps, wireless valves, X-ray tubes, rectifiers and a host of more recently developed apparatus of nuclear physics and nuclear engineering. The author then makes it clear that he did not intend to deal with the first problem and that the book would be entirely devoted to the applications of high vacuum.

The first chapter gives a summary of the fundamental facts of the kinetic theory of gases, in particular of those topics which are of direct consequence for high vacuum technique. It is based on the corresponding sections of Bloch's Kinetic Theory of Gases, published in 1926, even to the extent of borrowing whole sentences and paragraphs from it. This in itself is quite justifiable because Bloch's simple and clear treatment of the subject could hardly be bettered and our basic notions on the kinetic theory of gases have not altered in the last fifty years.

Chapter 2 deals with pumps, both of the mechanical and of the diffusion-condensation type. Many sections of this chapter especially those referring to earlier models are based on Dunoyer's excellent monograph on high vacuum technique published in 1924, but more recent developments such as oil diffusion pumps and fractioning pumps are also mentioned. Again, in Chapter 3, devoted to vacuum gauges, familiar passages from Dunoyer's book greet us at every step, but we are fortunately offered occasional glimpses of some newer types, *e.g.*, Penning gauges, bi-metal strip conductivity gauges, etc.

It is unfortunate that when he reaches Chapter 4, on vacuum plumbing, the author seems to have decided to stick entirely to Dunoyer and not to pay even lip service to any new fangled ideas. Had it been the other way round it might have been defensible. Important as the developments of pumps and gauges have been in the past 25 years all such equipment is now obtainable commercially. Thus, while it is necessary for the vacuum physicist or engineer to understand the working of these various instruments, he need not worry about details such as maintenance or repairs, as the supplier or manufacturer looks after that. But when it comes to the actual construction of a vacuum apparatus, to the choice of the right materials and components, to the tracking down and the elimination of leaks, it is then that he should be able to draw on the experience of others and that he expects to find guidance and helpful ideas in a book on the subject.

There is no doubt, that the advances in vacuum plumbing have been most spectacular in the last 10 years, the two chief reasons being the steadily increasing multiplicity and dimensions of apparatus for nuclear research and the vast amount of work connected with the separation of uranium isotopes by gaseous diffusion which eventually resulted in a large chemical plant built to high vacuum standards. It would not have been necessary for the author to delve into the declassified reports on this subject published by the U.S. and British atomic energy projects. A look at the last few volumes of the Review of Scientific Instruments, Journal of Scientific Instruments, Journal of Applied Physics or even a glance at the appropriate chapter of Dushman's

Vol.
1
1951

Scientific Foundations of Vacuum Technique would have given the author some useful and indeed indispensable material for the chapter on vacuum plumbing. It is inconceivable that with this information in his possession the author would have disposed of leak hunting in metal apparatus (having prescribed the electrical discharge method for testing glass apparatus) with the following words: 'If the apparatus to be leak-tested is made of metal, one can dip it into water, establish an overpressure of air inside and look for bubbles. If one cannot immerse it one covers it externally with soap solution.' There is no mention of the use of externally applied test gases in conjunction with Pirani gauges, ionisation gauges, mass spectrometers and all the variants and refinements of these methods; just water and air bubbles! One shudders at the vision of the big American K-25 diffusion plant immersed in a lake with scores of divers looking for bubbles.

The following three chapters deal with the degassing of glass and metal, the use of adsorbents and getters, and with 'Accessory Operations' which include items such as the measurement of pumping speed, gas micro-analysis, thin metallic films and even an item which could have been safely left out of a small book on vacuum technique, namely the cleaning of mercury. Just as through the rest of the book we have Dunoyer brought up-to-date after a fashion, and although there are some useful passages the whole arrangement of the subject matter is unsystematic and shows lack of care.

There are about 50 references to original papers distributed throughout the text and there is a short bibliography (8 works) at the end which is mainly notable for the happy play that the French typesetter was having with the spelling of English names. Thus we see that the author of a recent book on high vacuum technique is Yauvood, that of a somewhat older book on the production and measurement of low pressure F. H. Nemman and finally that great authority on high vacuum technique is referred to both in the bibliography and throughout the book as Dusham.

Looking at it as a whole M. Leblanc's 'La Technique du Vide' is a disappointing book. It has not got the broad outlook and the systematic handling of the subject which could make it into an elementary textbook on high vacuum. On the other hand it carries too much dead wood in the form of lengthy descriptions of old methods and antiquated apparatus and gives too little space to modern equipment and techniques for it to be really useful to a vacuum specialist, an aim which the author was striving for. It can only be hoped that either by revising this book or by commissioning a new book on the subject the Collection Armand Colin, will eventually have a monograph on high vacuum worthy of its reputation.

N. KURTI.

. . . . *of vacuum interest*

The Editor will be pleased to consider for publication in this column news items and personal notes of interest to vacuum workers.

Lectures

INDUSTRIAL APPLICATIONS OF HIGH VACUUM TECHNOLOGY. 12 lectures. To be held at the Northampton Polytechnic, St. John Street, London, E.C.1, from February 26th 1952 to May 27th 1952, at 7 p.m.

February 26th
The Design of Large-Scale Vacuum Plant
Lecturer : *T. S. Millen.*

March 4th
Leak Detection
Lecturer : *J. Blears.*

March 13th and 20th
Applications of High Vacuum Technology in the Optical Industry
Lecturer : *W. Zehden.*

March 25th
Applications of High Vacua in Metallurgy
Lecturer : *E. D. Malcolm.*

April 1st
Some Problems in the Use of Vacuum Evaporation for Industrial Purposes
Lecturer : *L. Holland.*

April 22nd
Freeze-Drying
Lecturer : *L. G. Beckett.*

May 1st and 8th
Glass-To-Metal Seals for High Vacuum Apparatus
Lecturer : *O. Adams.*

May 13th, 20th and 27th
High Vacuum Technology in the Valve Industry
Lecturer : *R. O. Jenkins.*

Fee for the course £1. Further details available from the Polytechnic Office, at the above address.

THE INSTITUTE OF METAL FINISHING incorporating the Electrodepositors' Technical Society, will hold a conference at the Grand Hotel Eastbourne from April 23rd to April 26th 1952. The lecture programme covers all aspects of metal finishing. One session will be devoted to vacuum techniques exclusively, as follows :

April 24th (Afternoon)
The Properties of Metallic Coatings Produced by Evaporation and Sputtering
Lecturer : Professor *S. Tolansky.*

Practical Considerations in the Application of Vacuum Coatings for Metal Finishing
Lecturer : *L. Holland.*

Lacquering to Obtain Brilliant and Metallic Lustre
Lecturer : *H. H. Vevers and G. E. Gardam.*

Provision is made for a discussion of the papers.

Miscellanea

DR. W. J. KROLL, the vacuum metallurgist who contributes to this issue informs us that he is coming to Europe this year and will visit this country on that occasion.

●

A TREATISE ON GAS BALLAST PUMPS which was being prepared by Wolfgang Gaede at the time of his death has now been edited and privately printed by Messrs. E. Leybolds Nachfolger. The booklet is written in German and will be reviewed in a future issue of ' Vacuum.'

●

THE BRITISH STANDARDS INSTITUTION is preparing a specification on the dimensions of Toroidal Sealing Rings, commonly known as ' O '-Rings and now widely used in vacuum equipment. Publication is scheduled early next year.

●

A READER OF THIS JOURNAL ASKED US whether we know of procedures providing for the preservation of butterflies and the like in vacuum. In spite of our efforts to give a helpful answer, we could not trace a single instance where vacuum was used as a means of preservation in that field. Is there a lepidopterist or entomologist among our readers who could provide the answer ?

Vol. 1 1951

ol.
1
951

VACUUM
CLASSIFIED ABSTRACTS

Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

Vol.
1
1951

Arrangement of Subjects

All abstracts published in this section are allocated to one or more of the subjects shown in the following list:

(I) General Science and Engineering

10 — VACUUM ENGINEERING — GENERAL
11 —
12 —
13 — ELECTRICAL SCIENCES
14 — MECHANICAL SCIENCES
15 — PHYSICS
16 — CHEMISTRY
17 —
18 —
19 — SCIENCES (other than the above)

(II) Vacuum Apparatus and Auxiliaries

20 — PUMPS
21 — GAUGES
22 — MEASURING PLANT — VACUUM
23 — INSTRUMENTATION
24 —
25 —
26 — MATERIALS
27 —
28 — APPARATUS COMPONENTS
29 — MISCELLANEOUS APPARATUS

(III) Vacuum Processing Techniques

30 — EVAPORATION
31 — CATHODIC SPUTTERING
32 —
33 — IMPREGNATION
34 — FUMIGATION
35 —
36 — DRYING
37 — METALLURGICAL PROCESSES
38 — DISTILLATION
39 — MISCELLANEOUS PROCESSES

(IV) Special Subsidiary Subjects

40 — GASES & VAPOURS
41 — FREEZE DRYING
42 —
43 —
44 —
45 — ELECTRONICS
46 —
47 — GLASS PROCESSING
48 —
49 —

Indices

Three indices are appended to each issue of abstracts: (1) A Cumulative Subject Index in front of the text, (2) A Cumulative Author Index at the back of the text and (3) a List of Periodical Publications quoted. Index (1) and (2) will show all abstracted information published up-to-date in the current volume of the journal.

Filing of Abstracts

All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract *e.g.*, 20/I as distinct from 20/II etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

Cumulative Subject Index

Subject — Abstract No.

I — General Science and Engineering — I

10 — VACUUM ENGINEERING (GENERAL) — 10

(For Abstract Nos. 1/I — 6/I see January Issue) (For Abstract Nos. 20/I — 27/I see April Issue)

Developments 55/I;
Distillation 1/I; 2/I; 22/I; 57/I;
Electrolytic Polishing 4/I;
Gas Evolution 6/I;
Gas Supply to Vacuum Systems 5/I; 59/I;
Gauges 58/I;
Gettering 23/I; 24/I; 25/I; 61/I;
Industrial Applications 20/I;
Metallurgy 21/I; 56/I;
Pumps 2/I; 3/I; 26/I; 27/I; 60/I;

13 — ELECTRICAL SCIENCES — 13

(For Abstract Nos. 7/I — 8/I see January Issue) (For Abstract Nos. 28/I — 37/I see April Issue)

Capacitors 28/I; 29/I; 62/I; 63/I;
Circuitry 33/I; 65/I;
Discharges 35/I;
Filaments 31/I; 32/I;
Hall Coefficient 37/I;
Insulating Means (Vacuum as an —) 7/I;
Lamps 36/I; 67/I;
Power Generators (High Frequency —) 34/I;
Process Recorder 64/I;
Resistance (Measurement of —) 30/I;
Resistors 8/I;
Thermocouples 66/I;

15 — PHYSICS — 15

(For Abstract Nos. 9/I — 14/I see January Issue) (For Abstract Nos. 38/I — 48/I see April Issue)

Crystals (Ionic —) 40/I;
Crystals (Metallic —) 12/I; 13/I;
Evaporation—General 77/I;
Evaporation Rates 11/I; 73/I; 74/I; 75/I; 76/I;
Filters (Optical —) 48/I;
Friction 68/I;
Gas—Solid Reactions 43/I;
Minerals 38/I;
Multiple-Beam Interferometry 46/I; 47/I;
Optical—General 69/I; 70/I; 71/I; 72/I;
Optical Methods of Measurement 45/I;
Organic Molecules (Measurement of —) 14/I;
Photo-Conductivity 10/I; 44/I;
Radioactivity 39/I; 78/I;
Radiography 9/I;
Surface Studies of Solids 41/I; 42/I;

16 — CHEMISTRY — 16

(For Abstract Nos. 15/I — 19/I see January Issue) (For Abstract Nos. 49/I — 53/I see April Issue)

Antibiotics 19/I; 49/I;
Biological Window 79/I
Blood Plasma 18/I; 50/I;
Fats (Animal —) 16/I;
Fermentation 17/I;
Food Preservation 15/I;
Hydrocarbons 81/I; 82/I; 83/I;
Micro-organisms 80/I;
Moisture Determination 84/I; 85/I;
Oils (Vegetables —) 16/I;
Oxygen Determination in Organic Substances 51/I;
Pharmaceuticals 52/I;
Vitamins 53/I;

Vol. 1 1951

Cumulative Subject Index

Contd.

Subject	Abstract No.
19 — SCIENCES (other than the above) — 19	
(For Abstract No. 54/I see April Issue)	
Powder Metallurgy	54/I;
II — Vacuum Apparatus and Auxiliaries — II	
20 — PUMPS — 20	
(For Abstract Nos. 1/II — 3/II see January Issue) (For Abstract Nos. 44/II — 51/II see April Issue)	
Charged Particle Accelerators (Vacuum Plant for —)	3/II;
Circulating Pumps	2/II; 88/II;
Diffusion Pumps	47/II; 48/II; 49/II; 84/II; 85/II; 86/II;
Ionization Pumps	45/II;
Molecular Pumps	87/II;
Piston Pumps	1/II; 44/II;
Rectifiers (Pumped —)	51/II;
Rotary Pumps	50/II; 89/II;
Toepler Pumps	2/II;
Vapour Lift Pumps	46/II;
21 — GAUGES — 21	
(For Abstract Nos. 4/II — 15/II see January Issue) (For Abstract Nos. 52/II — 60/II see April Issue)	
Bellows Manometer	9/II;
Developments (General —)	90/II;
Double Bellows Gauge	54/II;
Gauges (Various Types of —)	93/II;
Ionization Gauge	4/II; 5/II; 12/II; 13/II; 57/II; 58/II; 59/II; 60/II; 94/II; 95/II; 96/II;
Knudsen Gauge	6/II;
McLeod Gauge	7/II; 8/II; 92/II;
Mercury-Column Manometer	14/II;
Micromanometer	55/II; 91/II;
Philips-Type Gauge	11/II; 15/II; 56/II;
Pirani-Type Gauge	4/II; 10/II;
Resistance-Thermometer Operated Pressure Indicator	53/II;
Testing of Gauges	52/II;
22 — MEASURING PLANT (VACUUM) — 22	
(For Abstract Nos. 16/II — 32/II see January Issue) (For Abstract Nos. 61/II — 70/II see April Issue)	
Centrifuge (Vacuum type —)	108/II;
Electron Diffraction	18/II; 61/II; 62/II; 63/II; 97/II; 98/II;
Electron Microscope	17/II; 19/II; 32/II; 64/II; 65/II; 99/II; 100/II;
Mass Spectrometer	16/II; 27/II; 28/II; 29/II; 31/II; 66/II; 67/II; 68/II; 69/II; 70/II; 104/II; 105/II; 106/II;
Neutron Spectrometer	107/II;
Specimen Preparation	20/II; 21/II; 22/II; 23/II; 24/II; 25/II; 30/II; 101/II; 102/II; 103/II;
Telescope (Electronic —)	26/II;
26 — MATERIALS — 26	
(For Abstract Nos. 33/II — 34/II see January Issue) (For Abstract Nos. 71/II — 73/II see April Issue)	
Carbon and Carbon Products	72/II;
Gallium	73/II;
Nickel-Plated Iron	109/II;
Porcelain	33/II;
Potassium	112/II;
Properties of Materials (Vacuum —)	71/II;
Refractories	34/II; 113/II;
Tantalum	110/II; 111/II;

Cumulative Subject Index

Contd.

Subject	Abstract No.

28 — APPARATUS COMPONENTS — 28

(For Abstract Nos. 35/II — 38/II see January Issue) (For Abstract Nos. 74/II — 76/II see April Issue)

Cut-Offs	75/II;
Gaskets	38/II;
Leaks (Controlled —)	76/II; 118/II;
Motors (Electrical —)	37/II;
Traps	116/II; 117/II;
Valves (Mechanical —)	35/II; 36/II; 74/II; 114/II; 115/II;

29 — MISCELLANEOUS APPARATUS — 29

(For Abstract Nos. 39/II — 43/II see January Issue) (For Abstract Nos. 77/II — 83/II see April Issue)

Crystalliser	82/II;
Gas-Permeability of Materials (Determination of —)	80/II;
Gases (Storage of Liquefied —)	43/II;
Leak Detector	41/II; 42/II; 78/II;
Liquefier (Gas —)	81/II; 119/II; 120/II;
Pressure Control	39/II; 40/II; 77/II; 121/II; 122/II;
Radon Purification Plant	83/II;
Vapour Pressure of Materials (Determination of —)	79/II; 123/II; 124/II; 125/II;

III — Vacuum Processing Techniques — III

30 — EVAPORATION — 30

(For Abstract Nos. 1/III — 21/III see January Issue) (For Abstract Nos. 49/III — 65/III see April Issue)

Alkaline Earth Layers	17/III; 54/III; 60/III;
Aluminizing of T.V. Tubes	7/III; 106/III;
Cleaning Methods	107/III;
Continuous Band (Coating of —)	62/III; 63/III; 108/III; 109/III; 110/III;
Crystals (Growing of —)	5/III;
Etching	6/III;
Graticules (Manufacture of —)	65/III;
Metals (Deposition of Common —)	64/III;
Metals (Deposition of Precious —)	15/III; 18/III; 105/III;
Metals (Deposition of ' Rare ' —)	101/III; 102/III;
Multiple-Layer Films	49/III;
Oxide Films	57/III; 61/III;
Photo-Sensitive Layers	16/III; 53/III;
Physics of Evaporation	20/III; 98/III; 99/III;
Plant (Various Designs of —)	107/III;
Plastics (Deposition of Metal on —)	1/III; 4/III; 105/III;
Properties of Films (Electrical —)	56/III;
Properties of Films (Optical —)	55/III; 96/III;
Reflection-Reducing Films	3/III; 94/III; 95/III;
Replica Techniques (Various —)	11/III;
Resistors (Manufacture of —)	19/III;
Shadow Casting	8/III; 9/III; 10/III; 12/III; 13/III; 21/III; 103/III; 104/III;
Sorption of Gases by Films	58/III; 59/III; 100/III;
Structure of Films	14/III; 97/III; 98/III;
Testing of Films	90/III;
Thickness of Films (Measurement of —)	50/III; 51/III; 52/III; 91/III; 92/III; 93/III;
Thin Films—General	2/III; 89/III;

31 — CATHODIC SPUTTERING — 31

(For Abstract Nos. 22/III — 23/III see January Issue) (For Abstract No. 66/III see April Issue)

Etching	23/III;
Thin Films—General	22/III; 66/III;

Vol. 1 1951

Cumulative Subject Index
Contd.

Subject	Abstract No.

33 — IMPREGNATION — 33
(For Abstract No. 67/III see April Issue

Wood	67/III;

36 — DRYING — 36
(For Abstract Nos. 24/III — 25/III see January Issue) (For Abstract Nos. 68/III — 70/III see April Issue)

Antibiotics	25/III;
Concentration of Liquids (Methods)	68/III; 69/III; 111/III;
Evaporators	70/III; 112/III; 113/III; 114/III;

37 — METALLURGICAL PROCESSES — 37
(For Abstract Nos. 26/III — 35/III see January Issue) (For Abstract Nos. 71/III — 79/III see April Issue)

Carbon Determination	29/III; 72/III;
Concentration of Metallic Salt Solutions	119/III;
Developments (General —)	71/III; 115/III;
Distillation of Metals	34/III; 120/III; 121/III; 122/III;
Furnaces and Ovens	28/III; 31/III; 32/III; 78/III; 79/III; 115/III; 116/III; 117/III; 118/III;
Fusion Methods (Vacuum —)	30/III; 31/III; 123/III;
Impregnation Procedures	74/III;
Lost Wax Process	26/III;
Oxygen Determination	30/III; 123/III;
Preparation of Metals	33/III;
Pyrolitic Processes	73/III;
' Rare ' Metals (Processing of —)	27/III; 35/III; 75/III; 76/III; 77/III;

38 — DISTILLATION — 38
(For Abstract Nos. 36/III — 42/III see January Issue) (For Abstract Nos. 80/III — 85/III see April Issue)

Centrifugal Stills	128/III;
Developments	36/III; 124/III;
Fat Extraction	40/III;
Fats (Distillation of —)	39/III;
Fractionating	37/III; 39/III; 85/III;
Molecular	38/III; 41/III; 80/III; 81/III; 82/III; 83/III; 84/III; 126/III;
Safety Devices	42/III;
Short-Path Stills	127/III;
Stills (Various types of —)	129/III;
Vapour Liquid Equilibria	125/III;

39 — MISCELLANEOUS PROCESSES — 39
(For Abstract Nos. 43/III — 48/III see January Issue) (For Abstract Nos. 86/III — 88/III see April Issue)

Carbon Deposition by Cracking	86/III;
De-Odorization	44/III;
Food Processing	45/III; 46/III; 47/III; 48/III; 88/III; 132/III; 133/III;
Thermal Analysis (Differential —)	87/III;
Tritium Target (Preparation of —)	130/III;
Vitreous Enamelling	43/III;
Voltalization	131/III;

Cumulative Subject Index

Contd.

Subject — Abstract No.

IV — Special Subsidiary Subjects — IV

40 — GASES AND VAPOURS — 40

(For Abstract Nos. 1/IV — 8/IV see January Issue) (For Abstract Nos. 23/IV — 31/IV see April Issue)

Analysis of Gases 1/IV; 6/IV; 26/IV; 42/IV; 43/IV;
Blending (See Filling, Mixing and Blending)
Circulating of Gases 5/IV;
Dew-Point Bubble-Point Apparatus 3/IV;
Evolution of Gases 8/IV;
Filling, Mixing and Blending 4/IV;
Flow 23/IV; 24/IV;
Ionization 41/IV;
Liquefaction 31/IV;
Liquified Gases (Storage of —) 7/IV;
Organic Vapours (Determination of —) 2/IV;
Oxygen Determination in Organic Substances 25/IV;
Permeation 27/IV; 28/IV;
Sorption of Gases 29/IV; 30/IV; 44/IV;

41 — FREEZE DRYING — 41

(For Abstract Nos. 9/IV — 13/IV see January Issue) (For Abstract Nos. 32/IV — 34/IV see April Issue)

Apparatus and Methods (Various —) 46/IV;
Blood 9/IV; 33/IV;
Culture Preservation (Mycological —) 45/IV;
Fermentation 12/IV;
Food 13/IV;
Heating Systems 34/IV;
Plants (Vegetable —) 32/IV;
Tissue 10/IV; 11/IV;

45 — ELECTRONICS — 45

(For Abstract Nos. 14/IV — 20/IV see January Issue) (For Abstract Nos. 35/IV — 39/IV see April Issue)

Charged-Particle Accelerators 16/IV;
'Clean-Up'—Phenomena 20/IV;
Counters (Radiation —) 47/IV;
Electron Source 48/IV;
Oxide-Coated Cathode 14/IV; 15/IV; 35/IV; 36/IV; 50/IV; 51/IV;
Thermionic Emission 15/IV; 17/IV; 37/IV;
Valves (Electronic —) 18/IV; 19/IV; 38/IV; 39/IV; 49/IV;

47 — GLASS PROCESSING — 47

(For Abstract Nos. 21/IV — 22/IV see January Issue) (For Abstract No. 40/IV see April Issue)

Glass-To-Metal Seals 21/IV; 22/IV; 40/IV; 52/IV;

Vol. 1 1951

I — General Science and Engineering — I

Abstract No. and References

10 — VACUUM ENGINEERING (GENERAL) — 10

Vacuum Technique — 55/I

United Kingdom. Progress in the field of vacuum engineering is reviewed generally. Recent developments in pump design, *e.g.* the three-stage fractionating diffusion pump using octoil as a fluid, are mentioned. Reference is made to the alphatron gauge and silicone lubricants. Much space is given to the description of vacuum techniques now established in the metallurgical industry, in particular to an account of titanium and zirconium production, *i.e.* the Arkel-de-Boer iodide process and Kroll's magnesium reduction process. The article closes with a brief discussion of vacuum melting and casting procedures.

Sommaire : Ceci est un article passant en revue quelques applications de techniques du vide dans le domaine analytique et quelques-unes des applications dans le domaine métallurgique.

Article by S. L. Martin *Int. Chem. Engng.* **31**, July 1950 314-317

Vacuum Metallurgy — 56/I

See Abstract No. : 115/III

High Vacuum Distillation — 57/I

See Abstract No. : 124/III

The Measurement of High Vacuum by Electrical Methods — 58/I

See Abstract No. : 90/II

A Greaseless Gas Flow Valve — 59/I

United States. The valve described here is used for the admission of gas samples to an evacuated system. It consists of a ' U '-shaped glass tube terminating in a sintered glass disc at either end. One is connected to a suction pump or alternatively to atmosphere, the other to the sample gas container. A third sintered glass disc fits into an opening in the wall of the manipulating arm of the ' U ' and connects to the evacuated system. The tube is filled with mercury, the level of which—in the closed position of the valve—lies above the opening leading to the evacuated system. When suction is applied the mercury column in the manipulating arm drops, thus facilitating the penetration of the gas from the storage container to the system.

Sommaire : Description d'un robinet simple permettant l'admission et le réglage d'échantillons gazeux dans un système évacué.

Note by R. E. Honig *Rev. Sci. Instrum.* **21**, Dec. 1950 1024-1025

Construction of an All-Glass Mercury Circulation Pump — 60/I

See Abstract No. : 88/II

Some Properties of Tantalum — 61/I

See Abstract No. : 110/II

13 — ELECTRICAL SCIENCES — 13

Metallised Paper for Capacitors — 62/I

United States. The following formula is given for the calculation of the volume occupied by a wound capacitor ignoring insulating margins, outer wrappings and housing :—

$$\frac{V}{C} = 72.9 \frac{d(d + t)}{e}$$

Where V = volume in cc., C = capacitance in microfarads, d = thickness of dielectric in mils, t = thickness of electrode metal in mils and e = dielectric constant of the dielectric. As an example, this formula is applied to the case of a capacitance C needed for a low voltage application of, say, 100 volts. A metal foil capacitor of this type requires two layers of 0.3 mil paper with 0.25 mil aluminium foil. On the other hand, if metallised paper is used, only one layer of 0.3 mil paper with a lacquer coat of 0.03 mil is required. The formula gives a volume ratio of the metallised to the metal foil design = 0.31 in this instance. A further advantage of metallised

I — General Science and Engineering — I
Contd.

Abstract No. and References

paper capacitors is discussed at great length, *i.e.* their self-healing property. In the case of the conventional design, it is necessary to use two or more sheets of paper between the electrodes as this is the only means of safeguarding against certain imperfections of the condenser paper, *e.g.* small holes, extremely thin spots, etc. In the case of the metallised paper capacitor, dielectrically weak areas of the paper will cause the thin metal coating covering these spots to oxidise, vaporise or melt into isolated globules, thus relieving these areas of excessive electrical stress. This makes it feasible to use single layers of metallised paper for low voltage capacitors and gives the opportunity of operating the dielectric at a higher electrical stress than in the case of foil capacitors. Finally, a brief reference is made to the history of metallisation processes beginning with the development of "Mansbridge foil." Modern processing methods are discussed in some detail.

Sommaire : Description des constructions, performances et modes de production de capacités au papier d'usage général.

Article by D. A. Mclean *Proc. I.R.E.* **38**, Sept. 1950 1010-1014

Metallised Paper Capacitors

63/I

United States. A table is given of the approximate reduction in volume of (2 μf) metallised paper capacitors of the type used in telephone systems over that of conventional paper foil units which is reproduced below :—

Rating Volts d.c.	*Approx. Volume Reductions of Unit*
125	75% ...
200	65%
400	45%
600	30%

The article deals essentially with the performance of such capacitors and the testing methods employed to check their performance properly. Paper foil units are ordinarily tested by placing them on a voltage equivalent to $1\frac{1}{2}$—$2\frac{1}{2}$ times its rated value and determining the time passed before failure occurs due to short circuiting. Then, the application of the 5th power rule gives the means of estimating their service life at the rated voltage. This method cannot be used for metallised paper capacitors. Due to their self-healing property they could pass this test and still be unsatisfactory in service. It is important to keep the sparking inside the capacitor to a minimum as sparking causes noise in the telephone circuit. Therefore, a new method of testing these capacitors has been developed and is described here. The (2 μf) capacitors rated at 125 volts d.c. were placed in groups of eight on several test voltages, each group being in series with an electronic circuit which recorded the number of momentary break-downs. At 250 volts 65 break-downs were recorded in a period of 1,000 hours, at 200 the number was reduced to 19, and at 150 volts no breakdown at all was recorded in a period of 5,000 hours. In checking the performance of metallised paper capacitors on a.c. circuits reliance for detecting any deterioration was placed on the change of insulation resistance and power factor with time. Long storage periods of say two years and cyclic temperature tests covering the range of —40 to +50°C. did not produce any marked evidence of change in performance.

Sommaire. Traité concernant la construction, les performances et les procédés d'essai de condensateurs en papier, employés en téléphonie.

Article by J. R. Weeks *Proc. I.R.E.* **38**, Sept. 1950 1015-1018

A Metallised Paper Recorder

64/I

Germany. An endless band recording device is described which consists of a large-size roller as the drive, a pin point stylus as the scriber and a paper strip (up to 1,000 m.) covered with a thin zinc or cadmium coating (deposited *in vacuo*) as the endless record. These three elements form an electric circuit. In actual operation the pin point stylus burns the metal away, where it contacts the strip, and thus leaves a permanent track on the strip similar to an ink record on paper. The thickness of the metal coating is governed by the speed, at which the recorder is to operate. The correct thickness to use is best determined by the electrical resistivity of the coating rather than by physical measurement. With a thin deposit the strip can be moved with a speed as high as 10 m. per sec., *i.e.* phenomena of a duration not exceeding 10^{-4} seconds can still be recorded satisfactorily. This type of recorder has several advantages over the conventional design, *e.g.* low stylus friction and stylus mass, and high resolution capacity. A number of applications are discussed, particularly, in the field of telecommunications

Sommaire. Description d'un dispositif d'enregistrement, utilisant un ruban sans fin métallisé sous vide.

Article by A. Ortlieb *Elektrotech. Z.* **71**, 1.12.1950 653-656

A Reliable Pirani Vacuum Safety Circuit

65/I

See Abstract No. : **121/II**

The Use of Platinum Thermocouples in Vacuo at High Temperatures

66/I

United Kingdom. Pt/Pt13Rh thermocouples used in connection with thermal analyses of cobalt base alloys *in vacuo* at temperatures up to 1,625°C. showed a rapid change of calibration. The thermocouples consisted of wires 0.02 inch diameter fitted into a sheath of recrystallised refractories. One of the couples which had shown a greatly reduced e.m f. was examined spectrographically and it was found that, apart from other impurities being present, the platinum wire was contaminated with rhodium. An investigation was carried out to establish the proper cause of this deterioration taking into account the findings of Stauss and McQuillan. The former

Vol. 1 1951

I — General Science and Engineering — I

Contd.

Abstract No. and References

had proved that a very small percentage of rhodium in platinum varies the e.m.f. appreciably and the latter had shown that rhodium vapour tends to diffuse into the proximity of the platinum wire and is readily taken up by the same. It was observed that the rate of uptake was at its maximum as long as the wire contained no rhodium at all but with some rhodium present on the surface of the wire this rate was considerably reduced. On the other hand, evaporation of rhodium from the alloy wire had little effect on the e.m.f. As a result of this investigation a new thermocouple was made of the composition Pt1Rh/Pt13Rh. The performance of this thermocouple was greatly superior to that of those previously used. Twelve melts were made with the same couple showing no more than a calibration change of $\frac{1}{4}$–$\frac{1}{2}$°C. per run at the melting point of nickel.

Sommaire. Il a été trouvé que la contamination d'un thermo-couple Pt13Rh utilisé pour des procédés de fonte sous vide modifie les caractéristiques du couple d'une manière appréciable. Une modification de la composition du thermo-couple amène une amélioration.

Article by A. G. Metcalfe *Brit. J. Appl. Phys.* 1, Oct. 1950 256-258

Lamps Giving a White Light of Great Intensity

67/I

France. A definition of ' white ' light and its properties is given in the introduction to the article. The author then proceeds to review the design and performance characteristics of krypton and xenon discharge tubes examining at the same time the potential means of improving their output. Finally, a survey is made of the uses and applications of such lamps.

Article by M. Laporte *Le Vide* 6, Jan. 1951 927-935

Sommaire. Discussion de la construction et des caractéristiques de tube à décharge au xénon et au krypton.

15 — PHYSICS — 15

Dry Metallic Friction as a Function of Temperature Between 4.2°K and 600°K

38/I

United States. Little is known so far on metallic friction at low temperatures. The authors studied the friction between two unlubricated surfaces of the same metal in the temperature region of liquid helium. For the determination of the static friction coefficients a vacuum apparatus was used, details of which are given. The principle of a slider on an inclined plane was employed. The samples of the metal investigated were enclosed in a triple-walled glass cylinder, 35 inches long and 3.75 inches diameter, which could be tilted at any angle between the horizontal and the vertical. The outer jacket was kept at the temperature of liquid nitrogen to act as a cooled shield, the inner jacket was filled with liquid helium. The vacuum pumping unit employed consisted of a three-stage oil diffusion pump backed by a two-stage rotary pump. A liquid air trap was provided. All measurements were taken in a vacuum of 10^{-5}mm. Hg or better. The slider could be pulled back into the starting position by magnetic means. Details of the experimental procedure are given. It is stated that all measurements could be reproduced with great accuracy. In preparing the samples, care was taken not to decompose the oxide layers on the surface as this leads to conditions which should be termed adhesion rather than friction and would have little practical significance. Therefore, the samples were degreased and subsequently outgassed in a vacuum at a temperature not exceeding 250°C. In the case of lead, tin, zinc and hardened steel heat treatment could not be applied. Instead, the samples were subjected to ion bombardment in an atmosphere of purified hydrogen at a pressure of 0.1 mm. Hg. The authors came to the following conclusions: (*a*) Temperature coefficients of static friction of metals are rather small and for most of the metals positive, (*b*) the harder the surface, the smaller is the friction coefficient and at the same time its temperature dependence.

Article by I. Simon, H. O. McMahon & R. J. Bowen *J. Appl. Phys.* 22, Feb. 1951 177-184

Sommaire. Recherches sur le frottement entre corps métalliques secs, utilisant un appareil à vide pour l'établissement du facteur de frottement statique.

A Graphical Method of Determining Simultaneously the Optical Constants and the Thickness of Thin Metal Layers

69/I

France. In the first instance curves are obtained graphically which give the variations of reflection factors ' air/glass,' ' glass/metal ' and the transmission factors as a function of thickness for various values of optical constants. Using these and the measured transmission and reflection factors, curves are developed which give all pairs of values for refractive and absorption index. At the point, where these curves intersect, lies the pair of values to be determined. The thickness of the layer is determined by making a series of graphs for all values of $\delta = 4\ \pi d/\lambda$ where d is the thickness of the layer and λ the wavelength of the light. One set of curves gives the values of δ as a function of the absorption index for different values of the refractive index and one set of curves gives the values of δ as a function of the refractive index for different values of the absorption index. The correct value of δ can then be found by interpolation and the thickness calculated.

Note by D. Male *C. R. Acad. Sci. Paris,* 230, 3.4.1950 1349-1351

Sommaire. Description d'une méthode permettant la détermination graphique des constances optiques et l'épaisseur de couches minces métalliques avant préalablement mesuré la valeur du facteur de réflexion et de transmission en incidence perpendiculaire.

Phase Change of Light on Reflection at a Mica-Silver Interface

70/I

See Abstract No.: 96/III

Vol.
1
1951

I — General Science and Engineering — I

Contd.

Abstract No. and References

Achromatizing of Thin Films

71/I

Czechoslovakia. Complete extinction of reflection can only be achieved if more than one anti-reflection coating is applied to the optical component. One film will completely extinguish only one wave length of the incident light, two layers will extinguish two wave-lengths, three layers will extinguish three wave-lengths, etc. Multiple coatings showing the effect of extinguishing different wave-lengths of the incident light are called achromatized. This article gives an essentially mathematical treatment of achromatization phenomena. First, the case of a two-layer coating is considered. Finally, the author calculates full achromatization for three wave-lengths in triple coatings. This latter case, it is pointed out, could be of particular importance to the performance of optical instruments. For instance, if triple-coated lenses would be fitted to such instruments extinguishing the light of the wave-length of 4,600Å, 5,520Å and 6,440Å respectively, reflection would be reduced to almost zero just in those parts of the spectrum, where the eye is most sensitive.

Article by A. Vasicek *Optica Acta* 1, May 1951 20-25

Sommaire : Analyse mathématique des conditions d'extinction de réflexion lorsque l'on utilise des revêtements simples et multiples de réduction de la reflexion sur des éléments optiques.

Low-Reflectance Coatings and the Sensitivity of the Eye

72/I

South Africa. The reflection-reducing power of certain types of thin coatings reaches its maximum value at $n \times t = \lambda/4$, where n is the refractive index, t the thickness of the coating and λ the wave-length of the incident light. If a suitable refractive index is chosen, total extinction of reflection can be achieved. In practice, however, light sources are generally heterochromatic and thus a certain amount of reflection from adjacent wave-lengths remains. In addition, there is the further complication that the human eye is not equally sensitive to radiation in all regions of the spectrum. The author deals with the theoretical aspect of these conditions and comes to the conclusion that heterochromatic light makes little difference to the conditions established for monochromatic light. The presence of heterochromatic light can be disregarded in the respective calculations. But this applies only to the case of a single low-reflectance coating. Multiple-coating calculations are more complicated and are likely to produce less accurate results.

Article by J. H. van der Merwe & H. Verleger *Proc. Phys. Soc.* **64B**, Jan. 1951 76-81

Sommaire. Il s'agit d'une recherche sur la théorie de l'extinction de réflexion telle qu'elle est facilités en recouvrant des lentilles avec une couche de fraction de longueur d'onde et tenant compte de la sensibilité de l'oeil humain.

Vapour Pressure of Inorganic Substances. IV. Tantalum between 2,624 and 2,493°K

73/I

United States. This was determined by measuring the rate, at which a metal surface evaporates in vacuum. Values of ΔH_0° were calculated from the individual vapour pressures and showed no appreciable trend, the average value being 185.5 ± 0.3 kcal. By combining this value with free energy functions for solid and gaseous Ta, the following equation was obtained : $R \ln p = -(185.5 \times 10^3)/T + 3.7 \times 10^{-4} T - 8.4 \times 10^{-8} T^2 + 32.87$. (*Science Abstracts*)

Article by J. W. Edwards H. L. Johnston & P. E. Blackburn *J. Amer. Chem. Soc.* 73, Jan. 1951 172-174

Sommaire. La pression de vapeur du tantale est établie par la mesure de la vitesse d'évaporation d'une surface de tantale sous vide.

Vapour Pressure of Inorganic Substances. V. Zirconium between 1,949 and 2,054°K

74/I

United States. This has been determined by measuring the rate, at which a Zr surface evaporates into a vacuum. Values of ΔH_0° have been calculated from the individual vapour pressures and show no appreciable trend, the average value being 142 150 ± 350 cal/mole. By combining this value with free energy functions of gaseous Zr and with extrapolated values for the solid, the following equation is obtained : $\log P \text{ (atm.)} = -(31\,066/T) + 7.3351 - 2.415 \times 10^{-4} T$. (*Science Abstracts*)

Article by G. B. Skinner J. W. Edwards & H. L. Johnston *J. Amer. Chem. Soc.* 73, Jan. 1951 174-176

Sommaire. Détermination de la pression de vapeur du zirconium pare mesure de la vitesse d'évaporation d'une surface de zirconium sous vide.

The Evaporation of Titanium

75/I

See Abstract No. : 123/II

Evaporation and Mobility of Naphthalene Molecules

76/I

United Kingdom. The rates of evaporation of solid naphthalene into a vacuum were measured and it was found that their values varied for different crystal faces. Certain inconsistencies in the results of this study led to the suspicion that naphthalene molecules might be mobile over the surfaces of the solid phase and glass supports. To investigate these conditions a special experiment was conducted. A spot of solid naphthalene was placed in the centre of a glass disc of 20 mm. diameter and covered with a glass hemisphere of about 7 mm. diameter. Two such assemblies, facing in opposite directions, were mounted in a vacuum chamber. If lateral motion of evaporating naphthalene molecules occurred, a torque should be produced. Further, by forcing the molecules to migrate in a direction away from the axis of the suspension only, the torque moment should be increased and the difference of the two torques would give an indication of the mean distance travelled by evaporating mole-

I — General Science and Engineering — I
Cantd.

Abstract No. and References

cules along the surface. The experiment established beyond doubt that surface mobility exists. Using glass as a substrate the distance is estimated to be about 1 mm., in the case of graphite as a substrate the distance is estimated at 3 mm.

Sommaire. Recherche sur la vitesse d'évaporation et les phénomènes de mobilité de molécules de naphthalène, évaporées de l'état solide.

Letter by V. J. Clancey *Nature*, 166, 12.8.1950 275

The Life Time of Potassium Ions on a Tungsten Filament at 1,460°K

77/I

United Kingdom. A method is described of measuring the life time of potassium ions on a hot tungsten filament. A small rotor was placed in a vacuum chamber carrying a cup at the end of each arm. The rotor could spin at 1,500 c.p.s. driven by a rotating magnetic field. Tangentially to the path swept by the rotor tips a tungsten filament was placed with a cathode in its vicinity. In operation, potassium in the cups was heated up to evaporation temperature by induction means. Evaporated atoms hit the filament as the cups passed by. Potassium evaporates from a hot tungsten surface in the form of positive potassium ions. Therefore, the incidence of potassium atoms on the tungsten filament resulted eventually in pulses of potassium ions received by the cathode. Evaluating the experimental work described it was found that the time for the emission of positive ions from a tungsten surface at the temperature of 1,460°K is 3×10^{-5} sec.

Sommaire. La durée de vie d'ions de potassium sur un filament chaud de tungstène est étudiée et l'on calcule le retard d'évaporation de celui-ci.

Letter by T. H. Bull & D. G. Marshall, *Nature*, 165, 24.3.1951 478

A Geiger-Counter Mounting for Continuous Counting of Gas-Phase Radio Activity

78/I

United States. In a recent investigation of carbon dioxide-carbon reactions, an end-window Geiger counter was used for the continuous monitoring of the radio activity of $C^{14}O_2$ and $C^{14}O$ gases pumped over charcoal. The counter was sealed onto a small glass chamber but this arrangement was unsatisfactory as the counter window gave trouble. A thin window would not stand the stress of alternating filling and evacuating procedures, a thick window would affect the accuracy of the counter. The mounting of the Geiger counter, therefore, was changed. In the new arrangement the counter was sealed to the top of the male portion of a two-way hollow-bore stopcock which was incorporated into the vacuum system. This component was opened and flanged to fit the flange of the Geiger tube with the mica window cemented between them. The stopcock could be operated in any of the three following positions :—(1) The hollow part of the stopcock was connected with the system and permitted proper flow of the gases through the system. This position allowed for the continuous monitoring of the gases. (2) The stopcock blocked the flow of gases but allowed for the collection and counting of a static gas sample in the hollow part of the stopcock. (3) The hollow part of the stopcock was isolated from the system. In this position the system could be pumped and filled without subjecting the window of the counter to any mechanical stress.

Sommaire : Un compteur de Geiger est scellé sur un système à vide dans une position favorable pour le compte des gaz radioactifs circulant dans le système.

Note by F. Bonner & A. Weinstein *Rev. Sci. Instrum.* 21, Nov. 1950 934

16 — CHEMISTRY — 16

Biological Window

79/I

United States. Hitherto living tissue only provided the material for the construction of biological windows in living organisms. However, living tissue is usually non-transparent and it is proposed, therefore, to use non-biological materials suitable from the angle of transparency in combination with such materials as are tolerated by the biological tissue in contact with the window in position. It is suggested to use glass, plastics, etc. as a base material coated with a thin layer of tantalum, columbium or zirconium, all three of which are known to be acceptable to biological tissue. Not to reduce the transparency of the base material unduly, the metal coating should be very thin, *i.e.* of the type produced by evaporation *in vacuo*. Around the edge of the window the deposit could be somewhat thicker and roughened to adapt the window more readily for incorporation by the biological tissue after mounting.

Sommaire : Jusqu'à présent l'on utilisait des tissus vivants comme fenêtres biologiques. L'on propose d'employer des matières organiques métallisées (sous vide) avec du tantale, colombium ou zirconium.

A. H. Batchelder & California Research Corp. *U.S. Pat.* 2,517,523

Further Investigations on the Preservation of Mold Cultures

80/I

See Abstract No. : 45/IV

Lubricants Produced by Reactions in a Glow Discharge

81/I

See Abstract No. : 131/III

Vol. 1 1951

I — General Science and Engineering — I
Contd.

Abstract No. and References

Checking the Purity of Hydrocarbon Liquids by Means of Electron Diffraction

82/I

France. The authors examined light hydrocarbon compounds such as pentane, hexane or octane in the electron diffraction camera. It was the object of the investigation to ascertain whether a hydrocarbon liquid of this type composed of a chain containing but a few carbon atoms would—due to the grouping of the atoms in the molecule—produce a diffraction pattern of crystalline character. Details of the experimental work are given. After an initial run the technique of specimen preparation was altered to make sure that no impurities in the form of hydrocarbons of a higher molecular weight were present. In conclusion, the authors state that the electron diffraction patterns obtained from specimens of the short-chain type and prepared in accordance with the experimental techniques described, did not resemble those produced by an orthorhombic crystal structure.

Sommaire. Rapport sur des recherches utilisant des phénomènes de diffractions électriques pour l'étude de la structure du pentane, isopentane et quelques autres hydrocarbons.

Article by J. J. Trillat & C. Pierree
J. Rech. Centr. Nat. Rech. Sci. No. 13
1950
183-185

Vapour-Liquid Equilibria at Subatmospheric Pressures

83/I

See Abstract No.: 125/III

Methods of Estimating Moisture

84/I

United Kingdom. In assessing moisture content of a material three separate states of moisture have to be taken into account: (*a*) Free moisture, (*b*) physically bound moisture and (*c*) chemically combined elements of water. Free moisture is comparatively easy to deal with, physically bound moisture presents a more serious problem as the material will not liberate all moisture even if heated to a temperature well above that of boiling water or, if subjected to a desiccating agent or vacuum treatment. Chemically combined elements of water can only be driven off at the expense of actually decomposing the processed material. In actual fact, therefore, an organic colloidal system subjected to increasing temperature will release free moisture completely but physically bound moisture only gradually. Chemically bound elements of water will be gradually released at the same time but an increasing amount of the tested material will be lost due to decomposition. A survey is made of all available oven methods:—

(1) The water oven at appr. 210°F.
(2) The air oven at 212°F.
(3) The air oven at 221°F.
(4) The air oven at 239°F.
(5) The air oven at 248°F.
(6) The Brabender oven at 266°F.
(7) The Carter-Simon oven at 311°F.
(8) The Chopin Laboratory Tester at 392°F.

Vacuum oven methods are ordinarily an adaptation of air oven methods working at appr. 212°F. They effect a rapid initial release of moisture but the actual drying proceeds comparatively slowly. Vacuum methods give generally better results than methods working at atmospheric pressure. They offer definite advantages where the test material is liable to oxidation by air but do not suit materials with a high content of volatile matter. De Bruyn's method is mentioned.

Sommaire. Etude des méthodes existantes pour la détermination du degré d'humidité de substances donnant entre autre un tableau détaillé des mérites et défauts des méthodes connues utilisant un four de séchage.

Article by A. H. Ward
J. Inst. Fuel **24**, Jan. 1951
16-19

The Rate of Evaporation in the Determination of Water

85/I

United Kingdom. The determination of water by oven methods is an established procedure but it is laborious and not fully explored yet. The article gives particulars—mostly in tabulated form—of 13 different tests conducted with the aim of assessing a number of factors which influence the accuracy of their results. Such factors are:—the state of division of the substrates, the ratio of absorbant to substance, the amount of water used to mix absorbant and substance, pressure, time and temperature of heating, size, shape and material of the dishes used, etc. Vacuum oven methods showed as a rule a higher rate of evaporation than steam oven methods. This is attributed to the effective withdrawal of the moisture vapour from the oven by the pump.

Sommaire: Des recherches ont été faites sur les divers facteurs influençant les résultats de la détermination du degré d'humidité de substances au four.

Article by T. J. Mitchell
Chem. & Industr.
25.11.1950
751-753

II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

On the Method of Determining the Best Operating Condition of a Fractionating Oil Diffusion Pump] 84/II

Japan. The best operating conditions of Hickman's pump are usually indicated by the appearance of an oil ring above the boiler jet. The authors show that the oil ring corresponds to the highest final vacuum only if the fore-vacuum is satisfactory (1 to 2×10^{-3} mm. Hg) and suggests that measurement of the oil temperature is more reliable. On plotting oil temperature against current, the point of inflection of the curve corresponds to that current which gives the highest final vacuum and thus the best operating conditions.

(*Science Abstracts*)

Sommaire. Commentaires sur les méthodes usuelles de la détermination des meilleures conditions de fonctionnement de pompes à diffusion d'huile fractionnantes.

Paper by T. Hibi & K. Isikawa *Sci. Rep. Res. Inst. Tohoku. Univ.*, A1, Oct. 1949, 261-265

Reverse Separation in Fractionation Pumps 85/II

United States. It has been observed that multi-stage fractionating oil diffusion pumps did not produce the bottom pressure they were designed for, unless a very cold trap was used. A thorough investigation of this condition is reported in this paper. It has been found that a multi-stage pump run with a mixture of Octoil and Octoil-S in a proportion of 50 : 50 mole % gave a concentrate of 72.5 mole % Octoil deposited at the fine end of the pump and the vapour cruising in that area contained 94 mole % Octoil instead of chiefly Octoil-S as expected. In other words, the lighter constituents of a mixture tend to travel towards the mouth of the pump rather than to the fore-pressure end. The pump, designed to give a separation of more than 4 theoretical plates, actually performed at a rate of almost 4 negative plates. The phenomenon of reversed fractionation was demonstrated particularly clearly, when a pump running at the ultimate Octoil-S vacuum was charged with an amount of butyl phthalate which, if uniformly distributed throughout the pump, would have formed a 5% solution. As a result the pump performance deteriorated to a degree equivalent to the admixture of 27% butyl phthalate to the fluid in the boiler The above considerations are of practical importance only, where extremely high vacua are required, but they accentuate the importance of pre-purifying all pump fluids and operating under conditions preventing all contamination.

Sommaire. Cet article étudie le phénomène de la séparation dite ' renversée ' dans des pompes à huile fractionnantes. Les vapeurs des composants légers ont tendance à s'accumuler sous des conditions de vide poussé du côté haut vide de la pompe.

Article by K. Hickman *Rev. Sci. Instrum.* **22**, March 1951 141-146

An Air-Cooled High Vacuum Pump 86/II

Sweden. A mercury diffusion pump is described, the design of which dispenses with the conventional means of cooling the walls with water and relies on the cooling effect of the surrounding air by convection. It is claimed that the pump can attain vacua of the order of 10^{-1}mm. Hg against backing pressures as high as 20 mm. Hg. A.o., it is suggested to use the pump in cases, where small vessels have to be repeatedly evacuated.

Sommaire. Présentation d'une pompe à diffusion de mercure refroidie par air et qui atteint un vide de 10^{-4}mm. Hg avec un vide préliminaire de 20 mm. Hg.

Article by D. van Reis *ASEA Journal* **23**, Jan. 1950 17-19

The Design of Molecular Pumps 87/II

United States. A theoretical treatment is given of molecular pumps based on kinetic principles which yields formulae for compression ratios and pumping speeds simple enough to be readily applied to practical pump design. The pumping action of a molecular pump is initiated by the transmission of motion from a moving surface to molecules of the surrounding gas The effectiveness of this action is limited by the diffusion of gas molecules in the opposite direction due to the pressure gradient established in that direction, by the induced flow. First, the ideal case is considered, *i.e.*, a pump with a pumping channel of constant cross section and no leakage between the higher and the lower pressure region. Finally, formulae are given catering for the practical case of pumping channels which taper in the direction of the pumping action and for the presence of a leakage path of the type mentioned. The performances of a Friesen and a Gaede multi-stage pump are calculated using these formulae and compared with measurements taken during an actual run.

Sommaire. Développement de formules suffisamment pratiques pour servir de guide au constructeur de pompes moléculaires.

Article by R. B. Jacobs *Rev. Sci. Instrum.* **22**, Feb. 1951 217-220

Construction of an All-Glass Mercury Circulation Pump 88/II

United Kingdom. An all-glass mercury circulation pump has been constructed for the continuous removal of the distillate from a laboratory-size vacuum still, to be used in instances, where danger of contamination by the lubricant or other reasons make it undesirable to employ a mechanical pump. It has no moving part, is simple to build and its principle of operation resembles in some ways that of the Spregel pump. The pump consists of 5 major components :—A receiver at the top of the apparatus, a separator at the bottom, a down-take and an up-take capillary connecting the two and a controlled air leak fitted to the up-take capillary at a distance of 10-20 cm. above the separator. The distillate collects in the receiver which is incorporated in

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

the vacuum system of the still. The mercury circulating through the receiver traps the distillate and carries it to the separator where it emerges and floats on the surface exposed to the atmosphere. The circulation of the mercury is initiated by the vacuum itself. As soon as the receiver is connected to the still, the mercury will rise in both capillaries. Admission of air through the air leak will lift the mercury in the up-take capillary and deliver it into the receiver above the level of the mercury in the down-take tube. The dimensions of the apparatus given in the article allow for a throughput of 500-600 ml/hr.

Sommaire. Pompe à circulation de mercure, entièrement en verre fonctionnant selon un principe similaire à celui d'une pompe de Sprengel.

Article by H. Kerner *Chem. & Industr.* 13.1.1951 24

Vacuum Pumping Method with Mechanical Oil-Sealed Pumps

89/II

United States. The oil of mechanical vacuum pumps tends to become contaminated with water vapour or water condensate. One means of purifying the oil is the provision of a dry-ice dehydrating trap in the vacuum line leading to the pump. It is suggested in this patent to substitute dry-ice traps with an arrangement, whereby a source of supply of dry air is fitted to the pump near the exhaust end which delivers dry air or gas into the pump absorbing the water vapour and preventing its condensation in the rotor housing. The quantity of air fed into the pump varies with the size of the latter. The minimum required is that which reaches saturation after having taken up all the vapour present.

Sommaire. Il est décrit un dispositif, monté sur des pompes à palette pour éliminer les vapeurs condensables et les produits de condensation dans l'huile de la pompe, lequel rendrait inutile l'emploi de pièges réfrigérés.

F. A. Knox U.S. Atomic Energy Commission *U.S. Pat.* 2,551,541

21 — GAUGES — 21

The Measurement of High Vacuum by Electrical Methods

90/II

United Kingdom. The principle, the theory and some applications of all types of modern electrically operated high vacuum gauges are discussed. In the introduction, brief reference is made to the history of their development. A.o., it is mentioned that Morgan was the first to realise (in 1875) that the colour of an electrical discharge in an evacuated vessel is a function of the prevailing pressure. There are two major classes of electrically operated instruments: (a) The Pirani and thermocouple gauges and (b) the discharge tube and ionization gauges. The former actually records the pressure-dependent changes of thermal conductivity of the gas surrounding an electrically heated element and the latter directly records purely electrical phenomena, typical of pressure conditions. The following gauges are dealt with in great detail: The Pirani gauge in its original form and the modifications made to it by Hale and Campbell, the thermocouple gauges with reference to Kovalenko's research work, discharge tube pressure indicators and both types of ionization gauges, *i.e.*, the Philips-type cold-cathode gauge and the hot-triode thermionic valve type. Finally, the alphatron gauge is mentioned and the Knudsen gauge, the latter being the only instrument facilitating pressure measurement independently of the nature of the gas present in the evacuated vessel.

Sommaire. Etude de toutes les jauges électriques pour haut vide existantes.

Article by F. Wade *Electronic Engng.* **23**, Jan. 1951 30-34 & Feb. 1951 44-48

A Miniature Pressure-Recording Device

91/II

United States. A manometer was required sufficiently small to permit its introduction into the circulatory system for use in connection with the medical technique of intracardiac-catherization. The design described and shown in a sketch has a maximum diameter of 3 mm. and a metal tip 12 mm. long. It consists essentially of a piston made from brass, 0.6 mm. diameter, carrying a plate of 2.1 mm. diameter at one end and a small piece of soft iron at the other. The piston is held in position by a steel spring and activated by the pressure exercised on the end plate. The soft iron end of the piston acts as the armature of a differential transformer of the Wetterer design. Its position determines the relative coupling of the transformer sections. Details of the electrical circuit required for pressure indication are given. Overall performance of the manometer is summarized as follows: (1) natural frequency—1,000 c.p.s. in fluid, (2) damping ratio—0.34, (3) maximum sensitivity—50 mA/100 mm. Hg, (4) range of linear response—50 to +250 mm. Hg.

Sommaire. Détails d'un micro-manomètre utilisé en rapport avec la cathérisation intracardiaque à but diagnostique.

Note by O. H. Gauer & E. Gienapp *Science* **112**, 6.10.50 404-405

A McLeod Gauge with an Electrical Contact

92/II

United States. In the case of air-pressure operated McLeod gauges it is necessary to keep the mercury in the closed capillary at precisely the level required, whilst a reading is taken from the mercury column in the open capillary. This requires a certain amount of skill and the arrangement described here has been designed to overcome the slight difficulty experienced generally in this connection. A tungsten wire is sealed through the top of the closed capillary reaching nearly down to the bottom of the same. Mercury rising in this capillary and touching the tip of the wire causes a relay connected to the wire to operate which in turn will activate an electro-magnet. As a result, the armature of the magnet gives an audible click. This gives the observer an

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

opportunity to concentrate on the open capillary and take a reading, when the click occurs. A second reading is taken on the sound of a second click, *i.e.*, when the mercury, allowed to descend, again passes the tip of the wire. The two readings usually agree within ½ mm. or better.

Sommaire. Description d'une jauge McLeod à commande par air qui facilite des lectures plus précises qu'avec le type de jauge conventionnel.

Note by C. Kenty *Rev. Sci. Instrum.* **22**, March 1951 217-218

Improvements In or Relating to Pressure Measuring Apparatus 93/II

United Kingdom. The pressure measuring device proposed in this patent is intended to serve as a continuously indicating instrument in continuously pumped systems and can be applied in the micron range of pressures. It consists of a mercury or oil diffusion pump sealed with its fine end to the evacuated system. This pump—with a capacity of 2-3 litres per second—is shunted by a throttling device. The front of the throttling device is joined by pipes to the back-pressure end of the pump and the back of the throttle to the fine pressure end of the pump. A ' U '-manometer filled with an oil of low vapour pressure is used to indicate the pressure drop through the throttle which is the same as the pressure rise through the pump. In operation, gas is drawn from the system through the pump, discharged to the throttle and then returned to the fine end of the pump. Once the gas flow through this circuit has reached a steady value the whole arrangement acts like a continuously indicating McLeod gauge, where the circulating stream of vapour represents the compressing agent in place of the mercury in conventional McLeod gauges.

Sommaire. Description d'un indicateur de pression continue, construit pour utilisation avec un système à vide dynamique.

Ministry of Supply *Brit. Pat.* 639,281

A High-Speed Ionization Gauge 94/II

Argentina. A sketch and a short description are given of an ionization gauge which can be fabricated in any laboratory and conforms to the characteristics of a high-speed ionization gauge as discussed by Blears in a previous publication.

Sommaire. Description d'une jauge à ionisation rapide, basée sur les données, récemment publiées autre part.

Letter by J. M. Goldschwartz *Le Vide*, 6, Jan. 1951 955-956

Ion Gauge 95/II

United States. The conventional design of an ion gauge provides for three concentrically arranged electrodes : The electron–emitting filament is in the centre surrounded by the positively charged grid. This assembly is encased by a second cylindrical electrode, the negatively charged ion collector. In this design ions formed in the space between grid and filament can reach the collector only by passing through the grid. As a result, ions repulsed by the grid show a tendency to bombard the filament. This condition produces the effect of reducing the life of the filament and the sensitivity of the gauge. In the design proposed in this patent the three electrodes are positioned and shaped in such a manner that a large part of the ions formed in the space surrounding the filament can reach the collector without passing through the grid.

Sommaire. Description d'une nouvelle construction de jauge à ionisation qui permet à un nombre important des ions formés dans l'espace filament-grille d'atteindre le collecteur, sans passer à travers la grille.

S. Bashkin & U.S. Atomic Energy Commission *U.S. Pat.* 2,548,283

The Physical Basis of the Residual Vacuum Characteristics of a Thermionic Valve 96/II

See Abstract No. : 50/IV

22 — MEASURING PLANT (VACUUM) — 22

Checking the Purity of Hydrocarbon Liquids by Means of Electron Diffraction 97/II

See Abstract No. : 82/I

Experiments with an Electron Gun Designed for Use in Electron Diffraction Cameras 98/II

See Abstract No. : 48/IV

A Small Electron Microscope 99/II

United States. Reduction of the resolving power of an electron microscope to an ultimate 100 Å makes it unnecessary to provide for a condenser lens and a reduction of its direct magnifying power to ×3000 - ×6000 makes it possible to shorten the column and reduce the size of the vacuum plant required for the instrument. A ' bench-size ' type of electron microscope developed on these lines is described in this article. The instrument is 30 inches long from base to top of the hood and has a diameter of 4¾ inches. The vacuum plant consists

Vol. 1 1951

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

of a silicone oil diffusion pump coupled with a mechanical forepump and a valve arrangement very much simpler than that of the conventional electron microscope. Details are given of the optical system, the lens energization (effected by permanent magnets), the provisions made for specimen introduction and control, the viewing chamber assembly and the power requirements. The volume of the entire microscope excluding pumping elements is about 1 litre. This volume is divided into two pumping circuits : One, consisting of the plate chamber and viewing chamber, represents about 70% of the space to be evacuated, the other consists of the specimen chamber and electron gun. The valves are arranged to cater essentially for the following two operations :—(a) insertion and removal of the specimen and (b) insertion and removal of the photographic plate. In the case of (a) some 0.02 c. c of air will penetrate into the chamber during the process but no more than 6 seconds are required to re-establish operational pressure after insertion of the specimen. In the case of (b) more air is admitted and 60 seconds are required to re-establish operational pressure. Initial pumping-down time is 25 minutes. A thermocouple gauge is provided for pressure measurement.

Sommaire. Détail de construction d'un microscope électronique de table.

Article by J. H. Reisner, & E. G. Dornfield *J. Appl. Phys.* **21**, Nov. 1950 1131-1139

Continuous Observations with the Electron Microscope of the Formation of Evaporated Films of Silver, Gold and Tin

100/II

See Abstract No. : 98/III

Electron Microscopy of Wet Biological Tissues by Replica Techniques

101/II

United States. Replication of organic materials in the dry state tends to give unsatisfactory results. This is largely attributed to the fact that the cells of plant tissue, etc. in the natural state contain water and in this —a stretched—condition will reveal more of their submicroscopic structure than in the dry state. The author reports on a wet replication method which has been applied to wood fibres, cambial and bacterial cells. The wet specimen is placed on a polystyrene or polyethylene film and a few drops of distilled water are added. The film carrying the specimen is then placed between two microscope slides, locked with a clamp and immersed into a bath of distilled water of 100°C. After 7 minutes the sandwiched unit is taken out of the bath, allowed to cool down to room temperature and the clamp removed. After drying, the specimen is taken off and the replica ready for shadow casting. Gold shadowed replicas, prepared in accordance with this technique, are of a quality superior to that of replicas from desiccated specimens. In order to facilitate the preparation of silica replicas, known for excellent contrast properties, an extremely tenuous film is produced on a microscope slide by dipping it into a 1% solution of polystyrene in benzene and allowing the slide to dry. A replica of the wet specimen is produced in this film by the method outlined above. The film is then cut into small sections. These sections are floated onto a water surface, transferred to specimen screens and lifted on blotting paper, where the polystyrene settles tightly whilst the water is absorbed by the paper. The sections are now ready for a second replication by the silica evaporation method and are placed in a vacuum chamber of an evaporation plant for final treatment. Such replicas give very satisfactory results in the electron microscope but the technique employed in preparing them is somewhat hazardous. For that reason a further method has been devised involving an additional replication step, details of which are given.

Sommaire. Description d'une technique de préparation de réplique de tissus végétaux qui rend inutile le séchage du matériel avant prise de l'empreinte.

Article by I. W. Fischbein *J. Appl. Phys.* **21**, Dec. 1950 1199-1204

Replica Techniques in Electron Microscopy

102/II

United Kingdom. The observations recorded here have been made in the course of a study of the various steel structures of a low-alloy steel obtained by heat treatment. At one stage of this investigation the micrographs had to be photographed at a magnification of $\times 2000$ for comparison with specimens examined under the optical microscope. Such a magnification of a micrograph corresponds to an area of $40\mu^2$ and it was found difficult to keep such a large area of the object free from artefacts, unless special precautions were taken. For instance, in the case of martensite specimens small white dots were noticed on the specimen after mounting in a low-temperature thermo-setting plastic. These were established as a secretion product of the bakelite and eventually mounting of martensite specimen had to be abandoned altogether. Negative formvar replicas were prepared using the conventional dry stripping technique. This was unsatisfactory as the formvar could not be detached without showing strain lines. Therefore, a thick collodion film was introduced as a temporary backing for the formvar replica. A 40% palladium gold alloy was used for shadow casting and proved as successful as pure gold in most cases. In a few instances the gold content had to be increased to 80% to facilitate complete removal from the specimen. Finally, it is stated that pre-shadowing increases the amount of fine structure resolved and may yield an ultimate resolution of formvar replicas of 150 Å as distinct from 200 Å, hitherto believed to be the optimum.

Sommaire. Progrès réalisés à la technique de prépartion de répliques de surfaces métalliques.

Article by A. W. Agar & R. S. M. Revell *Brit. J. Appl. Phys.*, **2**, Jan. 1951 8-11

A Method of Preparing Paste and Powder Specimens for the Electron Microscope

103/II

United Kingdom. In preparing paste samples for examination in the electron microscope care must be taken to avoid aggregation of particles. Two known methods of preparing paste samples are quoted. Neither of them is safe in this respect. The method described by the author provides for the dilution of the paste in a ratio of 1000 : 1 in a spraying composition containing about 50% alcohol and 0.02% gelatin. The diluted

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

sample is sprayed onto a specimen grid which is pre-coated with a supporting film and placed near a heat source in order to effect rapid drying of each drop before the next reaches the same area. At this stage the gelatin present will form a gel and thus prevent aggregation of the sample particles. Subsequent immersion of the specimen in distilled water of 50°C for two minutes removes the gelatin without affecting the structure of the sample, which is now ready for use in the electron microscope or for shadow casting. The same technique can be employed with minor modifications for the preparation of powder samples.

Sommaire. Méthode de préparation de spécimens en pâte ou poudre pour l'examen au microscope électronique

Article by F. Schofield *Brit. J. Appl. Phys.* **2**, Jan. 1951 18-20

Instrumentation (Several new Mass Spectrometers. . . . .) 104/II

United States. Various mass spectrometer designs suitable for monitoring continuous processes are described. Particular reference is made to a special adaptation of the conventional magnetic deflection type of instrument, fully discussed in a paper presented jointly by C. F. Robinson, H. W. Washburn, C. E. Berry and G. D. Perkins to the 5th National Conference of the Instrument Society of America. Further, two fundamentally different types are mentioned which are still in the development stage. Both are radio-frequency mass spectrometers. The first is known as the Bennett type (see abstract 69/**II**) the second as the ' omegatron or ion-resonant type. Here, electrons emitted from a filament form ions in a region where crossed radio-frequency and magnetic fields are maintained causing the ions to follow a spiral path. The ions of the mass to be detected, *i.e.*, resonant with the set frequency of the field, are spiralling outwards until they reach the collector.

Sommaire. Trois constructions de spectromètres de masse pour l'analyse de gaz sont discutées.

Note by R. H. Munch *Industr. Engng. Chem.* **42**, *Nov.* 1950 93A-94A

Construction of a New Combined Vacuum Furnace and Mass Spectrometer for Universal Use 105/II

India. The investigation of vaporised materials for instance the study of thermal ionization etc., requires mass spectrometer instruments which are designed to incorporate a furnace to serve as the ion source. A short review is given of mass spectrometer design development in general. None of the instruments known to the authors fulfils the particular requirements of collecting ions of the same mass at one place so that they can be measured separately from the others and for this reason the authors designed an instrument, details of which are given in this paper. Its essential components are :—the ion source, the accelerating slit, the magnetic field and the collector. The ion source consists of a graphite tube heated by a current of approximately 1,000 amps. The tube is situated inside the evacuated chamber and has a side tube of graphite or iron attached to it, the latter containing the substances to be investigated in the solid or liquid state. The vapours of these substances on entering the main furnace are thermally dissociated into atoms, ions and electrons. These dissociated products effuse through the accelerating slit in the form of a composite ion beam consisting of particles of all velocities obeying Maxwell's distribution law. Details of the accelerating slit, magnetic field and the collector are given. The thermal velocities in the region of the slit are of the order of 0.5 volt, therefore an accelerating voltage of about 30 is sufficient to produce a homogeneous beam. The magnetic field is produced by an electro-magnet and the collector consists of two Faraday cylinders, one for the direct and one for the deflected beam of ions.

Sommaire. Description d'un spectromètre de masse, employant un four à vide comme source d'ions pour l'étude de l'ionisation thermique à l'état vapeur.

Article by B. N. Srivastava & V. N. Saharia *Proc. Nat. Inst. Sci. India*, **6**, July-Aug. 1950 263-270

Gas Flow Through the Mass Spectrometer Viscous Leak 106/II

United States. There are two types of leaks used for the admission of gas samples to the mass spectrometer, the molecular leak and the viscous leak. The operation of the former has been fully explored and described before. The latter offers great advantages particularly in connection with the continuous monitoring of a gas stream as it facilitates the admission of gas from a high pressure region. There is little known about the performance of this leak. An approximate equation is developed in this article for the flow of a binary gas mixture through a viscous leak and its accuracy is checked by actual performance measurement. For the purposes of this investigation the inlet system is divided into three sections :—(a) the flow through the spectrometer, (b) the flow through the constriction and (c) the flow through the capillary on the high pressure side of the constriction. As the flow through the system is continuous the expressions found for (a), (b) and (c) can be equated. One can now find an approximate value for the partial pressure of each component of the binary mixture in the ion source of the instrument in terms of its partial pressure in the sample. The ratio of these two expressions gives one expression for the variation in the measured sample composition with sample pressure.

Sommaire. Etude théorique sur les performances de systèmes d'admission de gaz pour spectromètres de masse, utilisant une fuite visqueuse, que l'on distinguera d'une fuite moléculaire.

Article by R. E. Halsted & A. O. Nier *Rev. Sci. Instrum.* **21**, Dec. 1950 1019-1021

The Harwell Time-of-Flight Neutron Spectrometer 107/II

United Kingdom. Details are given of a 3.5 MeV linear electron accelerator mentioned in a recent progress report of the Harwell establishment and embodied in a time-of-flight neutron spectrometer. Neutrons, produced in bursts, are flying a known distance before they reach the detector. The time taken for the flight serves as a means of assessing their velocity and energy. The pumping plant consists of two 4-inch oil diffusion

Vol. 1 1951

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

pumps backed by a mechanical pump, providing for an operational vacuum of 5×10^{-6} mm. Hg. Pressure readings are taken with an ionization gauge. Neutrons are produced at a rate of approximately 2.5×10^{12} per second during each high-frequency pulse. A pulse lasts 2μ sec. and occurs every 1/200th of a second. Further details of design and performance of the neutron spectrometer are given.

Sommaire. Les caractéristiques et quelques données essentielles d'un spectromètre de masse à temps de vol récemment construit sont décrites.

Article by A. W. Merrison & E. R. Wiblin *Nature* **167**, 3.3.1951 346-347

Magnetically Suspended Vacuum-Type Ultracentrifuge 108/II

United States. Ultracentrifuges are used for the determination of the molecular weight or particle size of materials. There are two methods. One is known as the ' rate of sedimentation method ' and the other as the ' sedimentation equilibrium method '. The former usually only takes a few hours in each case, the latter may require a continuous run of several days, but is considered the more reliable of the two. It is an essential feature of experimental requirements that the speed of the ultracentrifuge is constant throughout and precisely known. It is further important that temperature effects are reduced to a minimum. The design of the centrifuge described in this article provides for the rotor, situated in an evacuated chamber, to be brought up to the required speed by a retractable shaft connected to an air turbine outside the evacuated chamber. After the initial acceleration the shaft is disengaged and the rotor kept floating in a magnetic field produced by a solenoid placed above the vacuum chamber. Under normal conditions, *i.e.*, if the vacuum in the chamber is equivalent to 10^{-5} mm. Hg or better, the rotor will loose speed at a rate not exceeding 0.1 rev. per second, per hour. To brake the speed of the rotor a gas, preferably helium or hydrogen, is admitted to the chamber. The pumping unit required consists of an oil diffusion pump backed by a mechanical pump.

Sommaire. Détails d'une ultracentrifuge à vide dont le rotor est suspendu magnétiquement afin d'éliminer les effets indésirables de température.

Article by J. W. Beams J. D. Ross & J. F. Dillon *Rev. Sci. Instrum.* **22**, Feb. 1951 77-80

26 — MATERIALS — 26

Plated Iron as a New Material in Vacuum Technique 109/II

Czechoslovakia. The question of a reliable substitute for nickel in electronics is complicated by the fact that nickel combines a great number of valuable and important characteristics which can only be obtained, if at all, by a carefully balanced composition of baser metals, especially low-carbon iron, alloyed with aluminium. Still, the best of these alloys has to be plated with nickel, in order to obtain the required surface characteristics of the nickel, for which there cannot be any substitute. The paper gives a very comprehensive and detailed survey of successful work in this direction in various countries. (*Science Abstracts*)

Sommaire. L'article étudie les usages de fer plaqué en place du nickel plein comme matériaux de construction en technique de vide.

Article by W. Espe *Slabopr. Obz.* **2**, Feb. 1950 31-39

Some Properties of Tantalum 110/II

United Kingdom. The effects were studied of deformation and annealing on the mechanical properties of tantalum. The experiments were carried out on tantalum rods, originally 0.1 inch thick. The deformation was produced by wire drawing techniques. It was found that a reduction in area of 90% produced a hardness of 200 D P N. Subsequent heat treatment for 30 minutes in a vacuum of 10^{-4} mm. Hg showed that a reduction of area produced by deformation lowered the softening temperature of the metal. In the case of 20% reduction in area, the softening temperature was 600°C, in the case of 80% reduction 400°C. Due to the accidental presence of leaks in the equipment used for the heat treatment it was noticed that tantalum absorbed gases in the temperature range of 1700-1900°C which resulted in the hardening of the material as distinct from hardening caused by deformation. Gas-hardened tantalum showed an increased electrical resistivity, whereas tantalum hardened by deformation showed no such increase. When heated to a temperature of 2200°C or above tantalum released the gases absorbed in the lower temperature range.

Sommaire. Etude des propriétés mécaniques du tantale, en particulier son effet durcissant (de gaz) dans la région de température où ce métal exerce un effet getter.

Article by R. H. Myers *Metallurgia* **41**, April 1950 301-304

Vapour Pressure of Inorganic Substances. IV. Tantalum Between 2,624 and 2,493°K 111/II

See Abstract No. : 73/I

Life-Time of Potassium Ions on a Tungsten Filament at 1,460°F 112/II

See Abstract No. : 77/I

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

Pure Oxide Refractories

113/II

United Kingdom. Alumina, magnesia, beryllia, zirconia, ceria and thoria are the oxides of the corresponding metals. They occur in the impure state as minerals and require a rather complicated treatment for their extraction in the pure state. The purity of the commercially available grades varies between 99% and 99.9% In addition these materials possess exceptionally high mechanical strength, extraordinary inertness to hydrogen, nitrogen and sulphurous atmospheres and very high melting points. Thoria for instance melts only at 3000°C. These properties make pure oxide refractories eminently suitable for use in connection with metallurgical processes carried out *in vacuo*. Crucibles made from these materials are used for instance in the refining of chromium or in the vacuum distillation of aluminium. Further, refractories of this class are used for the melting and casting of purified metals under a vacuum.

Article by D. Kirby *The Times (Industr. Suppl.)* 5, Jan. 1951 26-29

Sommaire. La production et les applications de matières réfractaires en oxyde pure sont discutée par rapport à leurs emplois pour des procédés métallurgiques sous vide.

28 — APPARATUS COMPONENTS — 28

A Greaseless Gas Flow Valve

114/II

See Abstract No. : 59/I

Multiple High-Vacuum Valve Unit

115/II

Australia. Details are given of a multiple valve unit which together with a single low-impedance valve of the conventional design caters for the four operations usually required to control the evacuation of a system :— (1) isolation of the diffusion pump from the system, (2) isolation of the diffusion pump from the rotary pump, (3) admission of a controlled amount of air to the system and (4) rough pumping of the system by the rotary pump only. (1) Is effected by the single valve unit, (2), (3) and (4) by the multiple unit. The latter consists of three valves assembled in a common housing and operated by a single camshaft fitted with a knob. The housing has four outlets : (a) to the mechanical pump, (b) to the fore-pressure end of the the diffusion pump, (c) to the vacuum system and (d) to atmosphere. A sectional view of the multiple unit is given showing the position of the valves in the case of operation (3).

Note by R. I. Garrod & R. A. Coyle *J. Sci. Instrum.* **27**, Aug. 1950 228-229

Sommaire. Une vanne multiple, à vide, est décrite laquelle par maniement d'une seule manette contrôle toutes les différentes opérations d'ouverture et de fermeture d'un circuit classique de pompage, sauf l'isolement de la pompe à diffusion du récipient pompé.

Device for Use with Water-Driven Vacuum Pumps to Prevent Water Sucking Back Into the Apparatus

116/II

United Kingdom. Fluctuations in water pressure can cause water jet pumps to suck back into the apparatus which is being evacuated. As a safeguard against this contingency water traps are usually provided. But the conventional type of such traps is not always suitable. In some cases they have to be of very large dimensions and, therefore, represent an appreciable addition to the volume to be evacuated. A short description and a sketch are given in this letter of a new design of a water trap. It consists essentially of a jar, about 5 cm. in diameter, filled with mercury to a height of about 1 cm. The jar is fitted with a rubber stopper at the top with two holes carrying two capillaries, one leading to the vessel to be evacuated and one to the pump. The former dips into the mercury, the latter is protected by a small tube which floats on the mercury. During evacuation, all water sucked back into the system collects in the jar on top of the mercury. This arrangement prevents at the same time water vapours which may be discharged back through the pumping line from reaching the evacuated system. Special provisions are made to stop mercury from being drawn into the pump, when the vacuum is broken.

Letter by P. R. Booth *Analyst.* 75, Sept. 1950 499-501

Sommaire. Présentation d'un piège à vapeur d'eau, empêchant le retour d'eau dans le récipient évacué, destiné à être utilisé avec des trompes à eau.

A Simple Trap for Use with a Water Pump

117/II

United Kingdom. A simple water trap is shown in a sketch and described in a note which is recommended for use where water jet pumps are employed to evacuate small apparatus. It consists essentially of a short piece of wide glass tubing fitted with a rubber stopper at either end. The rubber stopper at the upper end holds a 10 ml. pipette with a Bunsen valve attached to its taper. The valve slit is made with a razor blade on one side of the rubber tubing. The rubber stopper at the lower end carries a capillary which connects to the pump. Water sucked back into the line due to fluctuations of water pressure is stopped by the Bunsen valve and collects in the trap. When the pump recommences working, the trap is emptied automatically. Reference is made to P. R. Booth's design (see Abstract 116/II) and it is pointed out that the device described here will be sufficiently effective in most cases except where it is imperative to prevent water vapour reaching any drying agent present in the system.

Note by C. D. Geoghegan *Chem. & Industr.* 3.2.1951 94

Sommaire. Description d'un piège pour usage avec une trompe à eau prévenant le retour d'eau dans le système évacué.

Vol. 1 1951

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

Gas Flow Through the Mass Spectrometer Viscous Leak 118/II

See Abstract No. 106/II

29 — MISCELLANEOUS APPARATUS — 29

A Medium Capacity Helium Liquefier 119/II

United Kingdom. Details are given of the design and performance of a simple helium liquefier operating in accordance with the Joule-Thompson expansion method. The helium compressed to about 25 atmospheres is passed over activated charcoal for purification purposes and fed into a Dewar vessel for cooling at the temperature of liquid oxygen. The helium is then transferred to the main vessel housing the Linde-heat exchanger for actual liquefaction. The latter is 2.2 m. long and positioned inside a vacuum jacket together with the expansion valve and a collecting vessel of 1.2 litre capacity. The whole aggregate is refrigerated by liquid nitrogen. Liquid helium is removed from the collector by means of a vacuum insulated syphon. The liquefaction efficiency of the plant is about 0.15 and the liquid hydrogen consumption 1.5 litres per 1 litre of liquid helium.

Sommaire. Détails d'un liquefacteur d'helium fonctionnant selon la méthode d'expansion Joule-Thompson.

Article by G. R. Hercus & G. K. White *J. Sci. Instrum.* **28**, Jan. 1951 4-6

Design and Performance of a Joule-Kelvin Helium Liquefier 120/II

United States. A helium liquefier of the Joule-Kelvin type is described which has a capacity of 1 litre /hour. Its starting time is 15 minutes. The plant consists of two heat exchangers, a Joule-Kelvin valve and the liquid helium collector. The final heat exchanger, the expansion valve and the collector are enclosed by a vacuum jacket, the lower part of which is made of glass and the upper part of brass. The cooling medium is liquid hydrogen. The liquid helium is removed through stainless steel tubes by suction. Full details are given of the instrumentation required to control the process. Much emphasis is placed on the design considerations leading to the construction of the apparatus described. It is explained that much latitude is given to the designer with regard to the economy of such a plant as it is of greater importance in laboratory work to provide for a short starting time than for maximum economy in operation. The overall heat efficiency of the apparatus is very largely governed by design and performance of the final heat exchanger. In this case, a tube-in-tube arrangement was chosen. Starr's formulae were used for the assessment of the operational requirements.

Sommaire. Détails d'un liquéfacteur d'hélium Joule-Kelvin et des données de performances déterminant la construction d'un tel appareil.

Note by J. G. Dash M. W. Zemansky & H. A. Boorse *Rev. Sci. Instrum.* **21**, Nov. 1950 936-938

A Reliable Pirani Vacuum Safety Circuit 121/II

United States. A safety device is described which acts automatically in accordance with the pressure prevailing in the vacuum system. The device is claimed to have a long service life, to be free from ' chatter ' and to have a comparatively small operation differential. A Pirani tube connected to the vacuum system forms one arm of a bridge, the balance point of which is controlled by a potentiometer. On one side of the balance an a.c. signal produced on the grid of a miniature thyratron is out of phase with the voltage applied to the plate of the tube. Thus, the thyratron cannot fire. On the other side of the balance, the two signals are in phase and the tube will fire during each positive half cycle. The impulse is transmitted to a relay which operates two micro-switches. In a test under operational conditions the relay closed at a pressure of 75 microns Hg and opened at a pressure of 100 microns Hg.

Sommaire. Dispositif de sécurité actionné sous l'effet de pression et utilisant un tube Pirani comme élément de commande.

Note by W. Franzen & J. Horton *Rev. Sci. Instrum.* **21**, Nov. 1950 935

Design and Operational Characteristics of Cartesian Manostats 122/II

United States. Developments in the design of cartesian manostats are discussed beginning with the cartesian diver first applied to automatic pressure control by A. H. Holtzinger and J. W. Heyd in 1936. Modifications introduced by Plambeck and Pedlow are mentioned as well as the design initiated by Cook, Spadaro and Ritzer. Finally, two types only lately developed are quoted, one for use with chlorine gas and the other operating as a flow regulator. The theory of these apparatus is considered and results are given of experiments carried out to establish their sensitivity and capacity.

Sommaire. Etude du développement de la construction de manostats cartésiens.

Article by R. Gilmont *Analyt. Chem.* **23**, Jan. 1951 157-162

The Evaporation of Titanium 123/II

United Kingdom. This is a report on experiments carried out with the aim of determining the vapour pressure of titanium at its melting point. The investigation proceeded on the lines of the Langmuir method. Magnesium reduction titanium and iodide titanium was used in the form of filaments made from wire 0.6 mm. thick. Details of the evaporation apparatus used in the experiment are given and illustrated in a sketch. Titanium

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

wire of a length of 32 cm. was bent into a hair-pin shaped filament and mounted in the all-glass apparatus. The current was supplied from a battery through copper-to-pyrex vacuum seals and kept constant during each run. The evaporation was carried at a pressure of 10^{-5}mm. Hg and the temperature of the filament under test was measured by means of an optical pyrometer of the disappearing filament type. The evaporated material collected in a sleeve attached to the apparatus and was weighed after each run. All results given in this paper refer to the beta-phase of titanium. Table 1, reproduced below in abbreviated form, gives some of the results :—

Run Number	*True Temperature* (° *K*)	*Evaporation m* (*mg*)	*Time t* (*sec*)	*Rate of Evaporation* $Ex10^6$ ($gm.cm^{-2}$ sec^{-1})
F 7. A2	1808	8.5	458	4.832
F 8. A2	1729	3.7	690	1.071
F 8. A3	1658	4.6	3240	0.370
F 8. A4	1660	10.2	6900	0.390
F10. A2	1706	4.0	1063	1.052
F10. A3	1796	8.3	466	5.111
F11. A2	1804	10.3	452	6.001

The mass of titanium collected in the sleeve usually exceeded the mass lost by the filament by about 10%. This is attributed to the gettering property of the metal. Evaluating all experimental data the authors estimated the vapour pressure of titanium at its melting point *i.e.* at 2,000 ± 10°K, to be 1.2×10^{-2}mm. Hg.

Sommaire. Description d'expériences faites en vue d'établir la pression de vapeur et le point de fusion du titanium. Les vitesses d'évaporation à diverses pressions sont données.

Article by L. G. Carpenter & W. N. Mair *Proc. Phys. Soc.* **64**, B, Jan. 1951 57-66

Vapour Pressure of Inorganic Substances. V. Zirconium Between 1,949 and 2,054°K 124/II

See Abstract No. : **74/I**

Vapour Pressure of Inorganic Substances. IV. Tantalum Between 2,624 and 2,493°K 125/II

See Abstract No. : **73/I**

Vol 1 195

III — Vacuum Processing Techniques — III

Abstract No. and References

30 — EVAPORATION — 30

Plating by Vacuum Evaporation 89/III

United Kingdom. A survey is made of all aspects of metal deposition by the vacuum evaporation process. The basic principles of the technique are described, some technical data is given, *e.g.* the evaporation rates of various metals, and processing requirements of interest to the industrialist are discussed. Finally, practical applications are reviewed :—the production of astronomical mirrors, television tubes, sealed-beam headlights, quartz crystals for high-frequency apparatus, capacitors and Christmas decorations.

Sommaire : On examine toutes les applications industrielles des couches métalliques preparées par évaporation dans le vide.

Article by J. Lomas *Mach. Lloyd* **22**, 14.10.1950 87-92

A Hardness Tester for Thin Films 90/III

Germany. An instrument is described which is designed for testing the hardness of thin films, in particular, reflection-reducing films deposited on lenses. The coated side of the lens is exposed to the rubbing action of a plunger moving repeatedly across the lens face. The pressure exercised by the plunger can be varied. If the coating shows no defects after 20 rubbing actions of the plunger at a pressure of 2 kg/cm², the lens is passed as satisfactory. As the plunger is driven by an electrical motor, the operation of the instrument is fully automatic and facilitates the testing of the films in accordance with reproducible standards.

Sommaire : Description d'un instrument mécanique pour l'essai de dureté de couches minces, par example dépôts sur lentilles.

Note by Anon. *Foto-Kino-Tech.* **4**, March 1950 76

A Method of Determining the Thickness of Silver Deposited on Glass 91/III

United States. The method described here has originally been proposed by Gardner and Case. A small crystal of iodine placed on or near the silver layer to be measured will give off iodine vapour which reacts with the silver and forms silver-iodide. Eventually a transparent spot will appear on the silver layer, around which the silver is only partially converted into silver-iodide. If light is directed to this area it will be reflected in the form of coloured rings surrounding the transparent spot. The actual number of rings showing is a function of the thickness of the film. A table is added to this note giving the actual values of film thickness for various numbers of rings.

Sommaire : Description d'une méthode pour la détermination de l'épaisseur de couches d'argent sur du verre.

Note by L. S. Nelson *J. Chem. Educ.* **28**, Feb. 1951 103

An Interferometric Method for Accurate Thickness Measurements of Thin Evaporated Films 92/III

United States. The method described is based on the use of the Fabry-Perrot interferometer. This instrument possesses a calibrated screw facilitating direct readings and an adjustment mechanism which ensures proper positioning of the sample. A sodium lamp is employed as the light source with its characteristic D-lines serving as the standard of length. The separation of the two sets of fringes produced by the sodium light is approximately 500 wavelengths. When the separation is decreased by the amount M_x or increased by the amount M_y, the thickness T of the deposit can be ascertained from :

$$T = \frac{(M_x + M_y)}{4\ K}$$

where $\lambda = (\lambda_1 + \lambda_2)/2$ and $K = (\lambda_1\lambda_2)/2(\lambda_1 - \lambda_2)$. Full details of the method and its limitations are given.

Sommaire : Proposition d'une méthode de mesure de l'épaisseur de couches minces basée sur l'utilisation d'un interféromètre Fabry-Perrot.

Article by L. G. Schulz *J. Opt. Soc. Amer.* **40**, Oct. 1950 690-692

A Graphical Method of Determining Simultaneously the Optical Constants and Thickness of Thin Metal Layers 93/III

See Abstract No. : 69/I

Achromatizing of Thin Films 94/III

See Abstract No. : 71/I

Low-Reflectance Coatings and The Sensitivity Curve of the Eye 95/III

See Abstract No. : 72/I

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

Phase Change of Light on Reflection at a Mica-Silver Interface — 96/III

United Kingdom. The phase change of light reflected by silver films deposited *in vacuo* has been studied frequently, but usually from the point of view, whether its value varies with the thickness of the deposit. Faust was the first who studied the phase change of light reflected at a silver-coated surface and compared these measurements with those taken at an uncoated surface. But this method did not yield accurate results. The author describes a new method which is claimed to be more precise and consists of measuring the optical thickness of a mica specimen, 1/30 mm. thick, coated initially with a slightly transparent silver layer on both sides. Then, the mica is cleaved, silver deposited on the new surfaces and the optical thickness of the cleaved parts determined. The sum of the two new optical thicknesses differs from the optical thickness of the mica sheet before cleavage. This difference is a measure of the phase change at the silver layer on the newly formed faces. Spectroscopically pure silver was used in the experiments, evaporated from molybdenum crucibles. The results of the study do not fully agree with data published by Hass and derived from experiments carried out on opaque silver films. This is attributed to the fact that a chemical test of the silver deposited used in the present experiment showed that a small amount of molybdenum had been picked up during evaporation.

Sommaire : Aperçu d'une nouvelle méthode pour mesurer le changement de phase de lumière réfléchie par une couche d'argent mince déposée par évaporation, laquelle serait d'une haute précision. Il s'agit du développement d'une méthode originée par Faust.

Article by E. Eisner *Research* 4, April 1951 183-185

On the Production and Structure of Layers Condensed in Vacuo and Consisting of Aluminium-Silver and Aluminium-Magnesium Alloys — 97/III

Germany. The effects of heat on the structure of aluminium-silver and aluminium-magnesium layers condensed *in vacuo* have been investigated in the electron diffraction camera and by means of electrical resistence measurements. Certain conclusions are drawn from these observations explaining diffusion phenomena noticed in the deposits.

Sommaire : Etude de la structure d'alliages Aluminium-Argent et Aluminium-Magnésium produits par évaporation sous vide.

Article by A. Boettcher *Z. Angew. Phys.* 2, May 1950 193-203

Continuous Observations with the Electron Microscope of the Formation of Evaporated Films of Silver, Gold and Tin — 98/III

Canada. The structure of thin films of a thickness below a certain critical value is fairly well known today but less has been published on comparatively thick films. The authors refer to a paper compiled some time ago by the Physics Dept. of the University of Toronto reporting on an attempt to record all phases of film formation by preparing and examining a series of films of gradually increasing thickness. The present article reports on an investigation carried out on the same lines but using more refined means. Examination of silver, gold and tin layers seems to indicate that migration of evaporated atoms on the substrate leads to the formation of nuclei upon which subsequent atoms collect and form aggregates. These aggregates grow and gradually merge together. Very few, if any, new nuclei appear at this stage. Details are given of the experimental arrangement used, which facilitated the deposition of the metal film inside the electron microscope and the continuous observation of the actual deposition process.

Sommaire : Etude de la formation de structure et des changements de dépôts évaporés, faites au microscope électronique facilitant l'observation continue.

Article by T. A. McLauchlan R. S. Sennett & G. D. Scott *Canad. J. Res.* 28A, Sept. 1950 530-534

Evaporation and Mobility of Naphthalene Molecules — 99/III

See Abstract No. : 76/I

The Effect of Crystal Parameter on Hydrogenation and Dehydrogenation — 100/III

United Kingdom. The article gives experimental proof that gas-induced orientation of evaporated nickel films produces (110) planes as a metal-gas interface. The films used in the experiment were (*a*) of the orientated type deposited in an inert gas atmosphere at a pressure of 1 mm. Hg and (*b*) of the unorientated type produced in a high vacuum. Surface measurements were carried out by the B.E.T. method and the number of crystallographic sites was measured by means of hydrogen adsorption at liquid nitrogen temperature. Using the nickel films as catalysts for the hydrogenation of benzene it was found that orientated films are about seven times more active than unorientated films. In the case of dehydrogenation of cyclohexane, unorientated films show a higher activity than (partially) orientated films and this leads to the assumption that unorientated films expose, at least in part, (111) planes.

Sommaire : Etude des caractéristiques d'adsorption et désorption de films de nickel, orientés et non-orientés en vue des conditions particulières obtenues dans l'hydrogénation du benzene et la déhydrogénation cyclo-hexane.

Article by O. Beeck & A. W. Ritchie *Discuss. Faraday Soc.*, No 8, 1950 159-166

The Preparation of Thin Tritium Targets — 101/III

See Abstract No. : 130/III

Vol. 1 1951

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

Biological Window 102/III

See Abstract No. : 79/I

Electron Microscopy of Wet Biological Tissues by Replica Techniques 103/III

See Abstract No. : 101/II

Replica Techniques in Electron Microscopy 104/III

See Abstract No. : 102/II

The Deposition of Rhodium on Perspex 105/III

United States. In the course of a physiological study of eye movements it was necessary to apply a highly reflecting coating to part of a contact lens made from perspex. The right choice of a metal was difficult. No distortion of the lens could be tolerated, and the wear due to rubbing by the eyelid and the corrosive effects of the tear fluid had to be considered. Eventually, rhodium was chosen for reasons of its high reflectivity, chemical inertness and hardness. The rhodium was deposited on the lens by evaporation in a chamber evacuated to 10^{-5}mm. Hg by silicone diffusion pumps. Prior to actual evaporation the rhodium was plated onto a tungsten filament which was serving as the crucible. Precautions had to be taken to prevent (*a*) breakage of the filament and (*b*) excessive heating of the perspex lens, before deposition of the rhodium at the desired thickness was completed: (*a*) was found to be dependent on the thickness of the tungsten wire and (*b*) was counteracted by connecting the workholder solidly to the large base plate of the evaporator in order to facilitate rapid heat conduction. The heater current was 45 amps. and the actual filament heat 2000°C. Evaporation was carried out in short bursts of about 1 second each.

Sommaire : Notes sur les expériences faites concernant le dépôt de rhodium sur des plastiques, expériences requises pour des recherches opthalmologiques.

Letter by B. L. Ginsborg & O. S. Heavens *Rev. Sci. Instrum.* **22**, Feb. 1951 114-115

Metal-Backed Television Picture Tubes 106/III

See Abstract No. : 49/IV

A 72-Inches High Vacuum Evaporator and Its Glow Discharge Equipment 107/III

United States. A large coating plant is described for the treatment of searchlight reflectors of 60 inches diameter. Its vacuum chamber consists of a vertical cylindrical tank some 62 inches high, top and bottom of which are removable. The top carries the necessary fittings to hold the reflector and the bottom carries the evaporation sources. The latter are fixed to a trolley carrying at the same time the power plant required. Two mechanical fore pumps and five oil diffusion pumps are provided. Four of the diffusion pumps are connected to a common manifold and controlled by a vacuum valve which isolates the whole group of pumps during the glow discharge cleaning cycle. There are eight evaporation sources (filaments) which are connected in parallel and can produce an opaque coating in 20 seconds. For the evaporation of silicon-monoxide (protective) layers a tantalum boat is provided of the dimensions 9 × 4 × 0.008 inches. Details are given of the glow discharge equipment provided for cleaning operations. Before a decision was taken on this equipment various methods of cleaning had been tested. The results of these tests are given and have a bearing on methods employed in smaller coating plant.

Sommaire : Discussion de la construction et des performances d'un appareil à métalliser sous vide de grande dimension utilisé pour la fabrication de réflecteur de phares de 155 cm de diamètre. Il est spécialement rendu compte des méthodes de nettoyage utilisées.

Article by N. C. Benson G. Hass & N. W. Scott *J. Opt. Soc. Amer.* **40**, Oct. 1950 687-690

Metallised Paper Capacitors 108/III

See Abstract No. : 63/I

Metallised Paper for Capacitors 109/III

See Abstract No. : 62/I

Metallised Paper Recorder 110/III

See Abstract No. : 64/I

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

36 — DRYING — 36

High Efficiency Vacuum Method. Simple and Effective Concentration of Aqueous Solutions 111/III

United Kingdom. The apparatus described is of laboratory size and designed for the concentration of thermolabile aqueous materials under vacuum. It is made of glass and consists essentially of a 5-litre flask connected by means of 3 inches wide bends and extension pieces to a condenser with a cooling surface of 3 sq. ft. A 20-litre flask serves as the receiver. A jet water pump is used fed from high pressure water mains. Details of an experimental run with the 5% solution of a sodium salt of a common organic acid as test liquid are given below :—

Condenser Water (in)	15°C.	Vacuum	15 mm. Hg
Condenser Water (out)	25°C.	Rate of Distillation—	
Condenser Water (Rate of Flow)	60 gal/hr.	(at Beginning)	5.1 l/h
Internal Temperature of Distilling Liquid	30-32°C.	(at End)	3.5 l/h
Water Bath Temperature	65°C.	Mean	4.25 l/h
Water Pump (Rate of Water Flow)	180 gal/hr.	Temperature of Condensed Distillate	16°C.

Increasing the water bath temperature improved the distillation rate. Cooling the receiver with ice water in addition, raised the distillation rate still further and reduced the temperature of the distillate from 17.5°C. to 12°C.

Note by M. A. Phillips *Chem. Age*, **63**, 11.11.1950 681-682

Sommaire : Description d'un appareil simple pour la concentration sous vide de matériel acqueux instable.

Experimental Falling-Film Evaporator 112/III

United States. The plant described was built to investigate methodically the application of the evaporation process to the production of juices and purees from fruits other than citrus, for which this process is now firmly established. All essential parts were made from glass to permit observation of processing conditions in all stages. The evaporator tube was 150 cm. high and 7.5 cm. wide. Hot water circulating through a jacket surrounding the evaporator tube was used as a heating medium. The liquid to be concentrated was drawn by vacuum into the tube at the top and was caused to boil rapidly as it flowed down in a film on the walls of the tube. The vacuum was provided by a 2-stage oil diffusion pump. The operational pressure varied between 12—347 mm. Hg. The temperature in the jacket was usually maintained at some 60°F. above the boiling point of the processed material. The initial experiments were concerned with the concentration of apple juice. In these experiments, evaporation took place at the steady rate of 5.4 litres/hr. until the ratio of soluble to solid content of the processed material reached a critical value. Thereafter, the evaporation rate dropped sharply.

Article by L. H. Walker & D. C. Patterson *Industr. Engng. Chem.*, **43**, Feb. 1951 534-536

Sommaire : La construction et performances d'un évaporateur à film descendant sont décrites, lequel fut construit pour des recherches sur des matériaux autres que des pamplemousse.

Rotary Film Evaporator For Laboratory Use 113/III

United Kingdom. In laboratory work it is frequently necessary to concentrate small quantities of aqueous solutions at low temperatures in a rough vacuum. To counteract excessive foaming of the solution during the process a small rotary concentrator has been developed, details of which are given in this note. An electrical motor adequately geared and fitted with a hollow shaft made from stainless steel is mounted on a pedestal about 1 ft. high at an angle of 45°. By means of rubber tubing the shaft is connected to a glass flask which is filled to about $\frac{1}{3}$ of its volume with the solution to be concentrated and floats in a water bath. Driven by the motor the flask is rotating and sets up a circular stirring motion of the water in the bath which keeps the flask in a central position. The upper end of the shaft is connected to a water jet pump through a non-return valve. Evaporation rates of up to 700 ml/hr. have been achieved with this apparatus.

Note by S. M. Partridge *J. Sci. Instrum.* **28**, Jan. 1951 28-29

Sommaire : Description d'un évaporateur de laboratoire, destiné à éviter le moussage excessif en rapport avec la concentration de petites quantités de solutions acqueuses.

Frozen Concentrated Citrus Juice Process Relies on Instruments 114/III

United States. A new plant for the production of frozen concentrated citrus juice is described. Its main feature is the use of three-stage single-effect falling-film type vacuum evaporators operated with ammonia refrigeration compressors instead of steam. The processing speed of the installation is exceptionally high. The water content of the juice is reduced from 89 to 44% within 4 minutes. No more than 20 minutes are required to cover all production phases from the time the juice is extracted to the moment it is sealed in the can. A flow sheet of the process is given but no details of the vacuum equipment.

Article by J. L. Heid *Taylor Technol.* **3**, Summer 1950 19-22

Sommaire : Description d'une installation de concentration de jus de fruits tels que citron, orange, etc. comprenant des évaporateurs à 3 étages à effet simple et à film descendant.

Vol. 1 1951

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

37 — METALLURGICAL PROCESSES — 37

Vacuum Metallurgy — 115/III

United States. A short survey is given of potential and established uses of vacuum in metallurgy. The detailed drawing of a split-tube graphite resistor vacuum furnace is added.

Sommaire : Présentation d'un four à vide, du type à résistance de graphite sous forme d'un tube fendu (dessin). Un certain nombre de méthodes à vide courantes dans le domaine métallurgique sont citées.

Note by Anon
Engng. Min. J. **152**, July 1951 134-135

H.F. Induction Furnaces — 116/III

United Kingdom. Four high-frequency induction furnaces have been installed at the Associated Electrical Industries Laboratories. One of these furnaces is designed for sintering processes *in vacuo*. One rotary and two oil diffusion pumps are provided. The minimum pressure obtainable is 10^{-5}mm. Hg. The heater circuit of the diffusion pumps is controlled by a pressure-operated relay which closes the circuit when the pressure prevailing in the furnace is of the value of 100 micron or less and opens the circuit when the pressure exceeds 150 micron. The electrical equipment required for this installation is described in great detail.

Sommaire : Description d'une nouvelle installation de 4 fours à induction H.F. sans bobine. L'un d'eux est un four à vide pour le frittage de métaux. Il est donné des détails de cette installation.

Article by Anon.
Elect. Rev. 10.11.1950 729-731

A Quenching Furnace for High Temperature Studies of Small Specimens — 117/III

United States. A furnace was required to facilitate the investigation of phase equilibrium relations of the systems ZrO_2-TiO_2 and ZrO_2-TiO_2-SiO_2. Investigations of this kind have to be carried out at temperatures of the order of 2300°C. The use of platinum-rhodium resistance furnaces is not practical in this temperature region. The furnace developed for the purpose consisted essentially of a tungsten-wire heater element placed between two water-cooled copper electrodes. A tungsten disc for the specimen was placed inside the resistor element. The wire of the resistor was 0.1 inches thick. It was found that the heater element could be used for several runs before a noticeable deterioration took place. A current of approximately 700 Amps. was required to heat the specimen to a temperature of 2100°C. After admission of the specimen the furnace chamber was evacuated to a pressure of 10^{-2}mm. Hg by means of a mechanical fore pump. All pressure readings were taken with a McLeod gauge.

Sommaire : Description d'un four construit spécialement pour des recherches sur les relations d'équilibre de phase de système ZrO_2-TiO_2 et ZrO_2-TiO_2-SiO_2.

Article by H. G. Sowman & A. I. Andrews
J. Amer. Ceramic Soc., **33**, 1.12.1950 365-366

A Small High-Temperature High Vacuum Furnace — 118/III

United Kingdom. Details of a furnace are given, the main feature of which is the provision of a resistance-heating system coupled with a reflector which concentrates the heat generated on the specimen. Resistance-heating is usually employed only where comparatively low temperatures are required, but it facilitates easy control and precise reproduction of temperature values. Induction heating systems, used frequently today in production on an industrial scale, are often unsuitable for research purposes due to the bulkiness of the equipment and the inherent difficulties of control since the amount of heat developed depends on the nature of the processed material. Arc-heating, the other alternative, may cause the contamination of the specimen by the material of the electrodes. As can be seen from the table reproduced below, unusually high temperatures can be produced in the furnace described in this article. The specimen to be heated rests on a ceramic disc and is surrounded by the heating element, a vertical helix of thick tungsten wire, positioned between two water-cooled copper electrodes. The reflector is of cylindrical shape surrounding the helix. Its upper end is closed by a removable circular lid with a hole in the centre to allow an optical pyrometer to be sighted on the specimen. The reflector consists of tantalum sheet on the inside, cased-in by five spiral turns of molybdenum sheet and protected on the outside by a riveted cylinder of the same material. The vacuum plant consists of a 2-inch oil diffusion pump backed by a mechanical pump.

Performance of the Furnace

Volts	2.4	5.4	8.3	10.5	13.3
Amperes	24.5	34	41	46	54.5
Watts	58.8	183.6	340.3	483	724.9
°C.	850	1325	1650	1800	2000

Sommaire : Un four à vide de laboratoire muni d'un corps de chauffe à résistance et d'un réflecteur, concentrant l'énergie de celui-ci sur la charge, réalise des températures exceptionnelles.

Article by K. B. Alberman
J. Sci. Instrum. **27**, Oct. 1950 280-282

Chromium Salvage — 119/III

United States. To overcome the shortage of chromium and chromic acid supplies during the last World War two processes have been developed for the recovery of chromium containing ions from anodizing and plating baths. The first employs the principle of ion exchange and has been developed for the treatment of anodizing

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

baths, the second is an evaporation process which is designed to concentrate wash water used for the rinsing of plated objects. These solutions contain from 1 to 6 ozs. of chromic acid per gallon and in the case of full plating strength 30 to 50 ozs. per gallon. The concentration is carried out in glass-lined stills of four different sizes, i.e. 200, 300, 500 and 1,000 gallons capacity. The operational pressure is approximately 4 inches of mercury. A steam jet pump is used for the production of the vacuum. The processing time is about 8 hours.

Sommaire : Il est décrit un procédé de concentration de bain de chromage par une méthode d'évaporation sous vide.

Article by Anon. *Chem. Wk.*, **68**, 3.3.1951 21 & 23

Vacuum Treatment of Parke's Process Crusts on a Pilot Plant Scale

120/III

United States. Parke's process crusts consist of zinc, lead and silver. Encouraged by the successful application of vacuum distillation in other fields an attempt was made to separate Parke's process crusts by the same means. Zinc distills at a temperature of 800°C. At this temperature zinc has a vapour pressure = 241 mm. Hg, lead = 0.0553 mm. Hg and silver = 0.0000276 mm. Hg. These values were considered to lie sufficiently far apart to give good prospects of success. A modified Pidgeon retort was used heated externally by six silicon carbide elements. The charge was heated in a boat made from 1/16th sheet iron and a 20-gauge black-iron liner was inserted at the cold end of the retort to serve as a condensing surface. The pumping plant consisted of a 6-inch oil diffusion pump backed by a rotary pump. In a typical run, heat and pumps were switched on simultaneously. Pumping was continued until a pressure of about 10-30 micron Hg was reached. The heating was switched off when the desired temperature was obtained. Subsequently, the charge was allowed to cool down to 200°C., at which temperature the pumps were stopped and the retort was opened. Two types of crusts were used : (1) a pressed crust of high copper content which could easily be stripped off the liner in sheet form, (2) a liquated crust of comparatively low lead content which was less easy to remove from the liner. The residue contained approximately 95% of the silver originally embodied in the crust. The results of the experiment are given in the form of graphs. It is shown that zinc could be removed successfully at low temperatures and lead at somewhat higher temperatures. Hot charging reduced the total time of the production cycle to 4 hours.

Sommaire : Rapport de la marche expérimentale d'un appareil construit pour la séparation de la couche de Parkes, un composé de zinc, plomb et argent, par distillation sous vide. Le résultat de cet expérience est considéré encourageant.

Article by A. W. Schlechten & R. F. Doelling *J. Metals*, **3**, Apr. 1951 327-330

Refining Lithium by Vaporization at Low Pressure

121/III

Canada. Lithium is a metal which is likely to be in great demand in the near future. Crude lithium contains ordinarily 0.5% sodium but sometimes this percentage is higher. In the majority of cases lithium must not contain more than 0.003% sodium to be useful. It is necessary therefore to refine the crude metal. The experiments reported here give an account of refining lithium by means of vacuum distillation. A retort was used approximately 60 inches long and 9 inches wide. One end of the retort was placed in a furnace and the other end was connected to the pumping unit consisting of a mercury diffusion pump backed by a rotary pump for producing the low pressure required, i.e. about 0.04 micron Hg. A second rotary pump was provided for rough vacua. The condenser was of a cylindrical shape and divided into five sections by means of circular ridges with one collecting pan for each of the sections. Three runs were made :—the first at 800°C., the second at 700°C. and the third at 600°C. The temperature distribution in the retort was checked by means of thermocouples in ten different positions along the whole of the interior of the retort. The temperature was kept steady for a period of about 5½ hours. The heating was then switched off and the retort cooled down to room temperature before it was opened to atmosphere. The results of the experiments are given in tabulated form. One of the tables is reproduced below :—

Expt. No.	*Temperature of Vaporization (Thermocouple 2) (°C.)*	*Location of Refined Lithium and Its Sodium Content*			
		Compartment 1 of the Condenser	*Compartment 2 Condenser*	*Compartment 3 of the Condenser*	*Compartment 4 Condenser*
I	800°	—	—	0.002	0.002
II	700°	—	0.002	0.003	—
III	600°	0.001	0.001	—	—

Sommaire : Rapport sur le raffinage expérimental du lithium par distillation sous pression réduite.

Article by R. R. Rogers & G. E. Viens *Canad. Min. Metall. Bull.* **44**, Jan. 1951 15-20

Analytical Distillation

122/III

United Kingdom. Reference is made to a method which has recently been developed for the quantative separation of sodium from aluminium. It essentially consists of distilling the metal compound in a vacuum and determining the sodium in the distillate by the zinc-uranyl-acetate method. The method is claimed to facilitate the determination of a sodium content of more than 0.005% with a precision not attainable by the use of existing methods.

Sommaire : Brève note concernant une méthode récemment développée pour la séparation de sodium de l'aluminium.

Note by Anon. *Light Metals* **14**, March 1951 149-150

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

Determination of Oxygen in Zirconium by the Vacuum Fusion Method

123/III

United States. Hitherto only residue methods have been employed for the determination of oxygen in zirconium. These procedures are tedious and lengthy and in many cases not sufficiently accurate with regard to their results. This article reports on the use of the vacuum fusion method for the determination of oxygen in zirconium metal. Reference is made to a method reported by Walter and Derge for the determination of oxygen in titanium by vacuum fusion. In the case of zirconium certain modifications have to be made to the method in order to get reliable results. (1) To obtain the required dilution small zirconium samples should be used (0.05—0.10 grams) and a small quantity of iron (15—20 grams) should be introduced prior to the first zirconium sample. Improper dilution of zirconium with iron results in obtaining less and less oxygen on subsequent samples. (2) The reaction temperature should be in the range of 1850—1950°C. Lower temperatures tend to give poor results. (3) The addition of tin in amounts up to 25% appears to promote the reduction of zirconium oxides. The part tin is playing in this connection is not fully known but it appears to influence the fluidity of the charge. The zirconium samples should be wrapped with annealed iron foil to avoid contact with the graphite crucible. The results of the vacuum fusion method show good agreement with those of the chlorine method. One analysis takes approximately 20 minutes.

Article by J. K. Stanley J. von Hoene & G. Wiener *Analyt. Chem.* **23**, Feb. 1951 377-379

Sommaire : Description d'une méthode permettant la détermination de l'oxygène contenu dans du zirconium métallique.

38 — DISTILLATION — 38

High Vacuum Distillation

124/III

United States. An account is given of major developments in the field of high vacuum distillation during the year 1950. One important event of the year is the appearance of molecularly distilled monoglycerides of fatty acids. Commenting on vacuum dehydration in general, it is stated :—some 120,000,000 gallons of fruit juice have been concentrated for freeze-canning during that year, involving the removal of about 180 thousand million cubic feet of vapour. The manufacture of a soluble coffee product has been announced, produced at a water vapour pressure of 1 mm. Hg and a residual air atmosphere of less than 30 micron. Finally progress in the design of short path stills is discussed and work carried out in the field of molecular distillation. Particular reference is made to the publication by Green and Watt on the distillation of tocopherol bearing oils (see abstract no. 81/III).

Article by K. C. D. Hickman *Industr. Engng. Chem.* **43**, Jan. 1951 68-69

Sommaire : Etude des développements remarquables annuels dans le domaine de la distillation sous vide.

Vapour Liquid Equilibria at Sub-Atmospheric Pressures

125/III

United States. The separation of high boiling hydrocarbons from their mixtures requires the use of sub-atmospheric pressures to avoid decomposition. The present investigation aimed at the clarification of vapour-liquid equilibrium conditions in the case of higher molecular weight compounds and was carried out on a tetradecane-hexadecene system. A Colburn type equilibrium still was used. Details of the experimental arrangement are given. Runs were made for the following pressures : 760 mm., 400 mm., 200 mm., 100 mm., 50 mm., 20 mm., and 10 mm. Hg. All results are given in tabulated form. Conditions at 200 mm. Hg appear to approach those of the ideal Raoult law more closely than conditions prevailing during any of the other runs.

Article by R. R. Rasmussen & M. van Winkle *Industr. Engng. Chem.* **42**, Oct.-1950 2121-2124

Sommaire : Rapport sur une étude des conditions d'équilibre vapeur-liquide à des pressions sub-atmosphériques telles qu'elles se présentent lors de la séparation d'hydrocarbone à point d'ébullition élevé, de mélanges liquides.

Molecular Still

126/III

United States. A molecular still is described which has been designed on the lines proposed by Quakenbush and Steenbock. However, the glass rotor originally fitted to the still has been replaced by a stainless steel rotor running on ball bearings and fitted to the top of the distillation chamber. The stirrer attached to the rotor is made of stainless steel and generally of dimensions which will ensure minimum interference with the actual distillation process. A photograph and drawing of the assembly are given. The still has a capacity of 1 litre. The vacuum plant consists of a two-stage oil diffusion pump and a rotary pump. Pressure readings can be taken with a Pirani and a McLeod gauge. It is proposed to use the still for the distillation of certain natural products which have proved refractory to conventional distillation techniques.

Note by M. Priznar W. A. Wilt & F. C. Nachod *Analyt. Chem.* **22**, Aug. 1950 1065-1066

Sommaire : Construction d'un appareil à distillation moléculaire qui comprend un rotor en acier inoxydable et un agitateur dans l'alambic.

Short Path Vacuum Stills

127/III

United Kingdom. Little has been published so far on the ' pot '-type short-path vacuum still. In spite of certain limitations these stills have been found useful for distillation work on a small scale. Two designs are described in this article both made from glass with a capacity ranging from a few grams to over 100 grams per charge. One of the stills is designed to operate in a horizontal position. The distilling vessel consists of a standard pyrex tube 20 inches long and 3 inches wide, closed at one end. The condenser inside is placed in a

III — Vacuum Processing Techniques — III

Abstract Nos. and References

slightly sloping position and has a rim at the lower end to collect the condensate. The heater consists of two pyrex tubes assembled concentrically. The inner tube carries a winding of nichrome resistance tape to serve as the heater element proper with an input of about 250 watts. The outer tube merely provides a protective cover. An auto-transformer in the heater supply circuit facilitates control of the heater temperature to a precision of ± 2°C. The mounting and the removal of the heater does not interfere with the rest of the still assembly and clear visibility of the still content is maintained due to the transparency of the heating device. The still is suspended in a manner which facilitates degassing of the charge whilst the still is in a vertical position.

Sommaire : Un appareil à distiller sous vide, du type dit 'type à pot' de laboratoire est décrit.

Article by A. R. Gilson *Chem. & Industr.* 18.11.1950 739-740

Improvements In or Relating to the Heating of Rotating Work-Pieces by Electro-Magnetic Induction

128/III

United Kingdom. This patent utilises the eddy-current effect for the generation and control of heat in the rotating member of a centrifugal still. If an aluminium disc is rotated in a magnetic field a resistance torque is set up by the field and the energy required to overcome it is transformed into heat which warms the aluminium disc. The amount of heat developed in the disc depends on the speed of rotation and the strength and geometry of the magnetic field. In ordinary processing conditions this effect is considered undesirable, but in this instance it is used to provide an efficient source of heat. The application of this principle obviates the necessity of providing individual heater elements, which have to be kept at a temperature in excess of that to be attained by the vaporizing surface with the attendant danger of over-heating and decomposing the distilland. Various arrangements are described, which ensure that the temperature of the vaporizing surface of the still is maintained at a predetermined value.

Sommaire : Ce brevet concerne une méthode fournissant et réglant la chaleur des pièces tournantes d'appareils à distiller centrifuges, utillisant l'effet de courants Eddy.

A. B. White R. B. Jacobs & Distillation Products Inc. *Brit. Pat.* 635,436

Low Cost Still Removes Light Ends from Fatty Acids

129/III

United States. A brief description is given of a two-stage continuous distillation unit specially recommended for the processing of unstable heat sensitive feed stock. The two stages are separated by a vertical wall open at the bottom to allow for the passage of the liquid. The crude material is preheated, dehydrated, and then fed to the still. The light ends are removed under high vacuum by the outgoing steam. The pressure drop is minimised by the provision of large-sized vapour passages. Both these factors allow for operation at very low temperatures.

Sommaire : Une brève note sur un appareil à distillation continue sous vide à deux étages.

Note by Anon. *Chem. Engng.* **58,** March 1951 195-196

39 — MISCELLANEOUS PROCESSES — 39

Preparation of Thin Tritium Targets

130/III

United States. Thin tritium targets, the production of which are described here, consist of a tungsten disc bearing a zirconium layer which has absorbed tritium gas. The deposition of the zirconium layer was carried out by evaporation in vacuum. The zirconium to be evaporated consisted of 6 mil. and 10 mil. annealed wire, a bundle of which was placed on a tungsten ribbon stretched horizontally between two copper posts. The zirconium wires were fused to the ribbon by ' flashing.' To evaporate the zirconium the tungsten ribbon was heated to a temperature of 2,500°C. in a vacuum of 10^{-5}mm. Hg. The tungsten disc, on which the zirconium is to be deposited was fixed in a position about $1\frac{1}{2}$ inches above the evaporation source. Approximately 25 minutes were required to complete deposition of the zirconium. Special precautions had to be taken to ensure that the right proportion of zirconium to tungsten was used and that the tungsten discs were carefully cleaned beforehand. After completion of the deposition process the tungsten disc was placed in a vacuum system which incorporated a stainless steel trap containing the compound UT_3. Heating the trap decomposed the compound into uranium metal and gaseous tritium. At this stage the valve to the pumps was closed and the valve in the line connecting the trap and the vacuum chamber was opened. The disc bearing the zirconium layer was heated whilst the tritium gas was passing over it, to facilitate absorption. The tritium target and the uranium trap were then cooled down allowing the compound UT_3 to reform from the remaining tritium. The targets prepared in this manner consisted of zirconium deposits of a weight from 10 gm. per cm.^{2} to 500 gm. per cm.^{2} with a tritium content of the ratio of 1 tritium to 1 zirconium atom.

Sommaire : La préparation de petites anti-cathodes en tritium est décrite, laquelle comprend le dépôt de zirconium sur une anti-cathode nue, sous vide.

Article by A. B. Lille & J. P. Conner *Rev. Sci. Instrum.* **22,** March 1951 210-211

Lubricants Produced by Reactions in a Glow Discharge

131/III

United States. This paper reports on an investigation into the performance and potential uses of the voltolization process. The object of this process is to produce additive agents for lubricating oils by the thickening of lubricant materials in a glow discharge. The method is of particular importance in connection with the production of aviation oil. The processed liquid passes between electrodes which carry opposite charges in an

III — Vacuum Processing Techniques — III

Abstract Nos. and References

evacuated chamber. On applying a suitable voltage a glow discharge is established and the liquid is being recycled through this area until the desired change in viscosity of the material has been achieved. According to the observations made by the authors, a number of reactions take place concurrently, for instance, polymerization and/or condensation leading to a wide distribution of molecular weight, hydrogenation and dehydrogenation and finally a general destruction of polar groups such as hydroxyl, carboxyl and carbonyl. Details are given of the experimental equipment used. The actual pressure, at which the reaction took place, appeared to be of minor importance, but from the electrical point of view it was more economical to operate the plant at a pressure of about 120 mm. Hg than at 20 mm. Hg. The investigation covered various blends of fatty oils, waxes and petrolatums of mineral origin. The fatty oil votols, designated as aviation concentrates, were ordinarily votolised to a viscosity of either 370-400 or about 1,000 Saybolt secs. at 210°F. Concentrates of lower viscosity than 370/210°F. were not considered practical, and those above 1,000/210°F. proved difficult to produce. It was established in this investigation that high quality lubricants could be prepared using voltolised concentrates, but the process has certain disadvantages: (*a*) production costs are high and (*b*) voltolised additive agents have to be of a higher concentration than those of other types of additives in many cases. Finally the theory of voltolisation is discussed in detail.

Sommaire: Voltolisation, un procédé de polimérisation de lubrifiants à base de pétrole par une décharge lumineuse.

Article by R. S. Whiteley C. N. Kiberlin G. L. Matheson & R. W. Richardson *Industr. Engng. Chem.* **42**, Dec. 1950 2471-2479

O

Process of Preparing Coffee Extracts

132/III

United States. A process is described which yields a thoroughly soluble product preserving the natural taste and dietetic properties of coffee. Aqueous suspensions of ground roasted coffee beans are distilled in a rough vacuum at a temperature not exceeding 42°C. without refluxing. The distillation is continued until the weight of the distillate is about equal to that of the distilland. The remainder of the aqueous suspension is extracted by means of water at a temperature of about 90-95°C. This aqueous extract may then be concentrated to form a syrup. In the final drying process it should be blended with the distillate produced as described. This final process should take place in an atmosphere of nitrogen under reduced pressure. The patent also describes a method of producing a composite coffee product which, on solution in water, yields a beverage on the basis of milk, sugar and coffee.

Sommaire: Description d'un procédé de fabrication d'extrait de café et de boissons composées contenant de l'extrait de café, du sucre et du lait.

P. A. Bacot Soc. Immobiliere & Financiere du Parc *U.S. Pat.* 2,522,014

High Vacua in Cans

133/III

United Kingdom. Reference is made to a method mentioned in the 1949 Annual Report of the Fruit and Vegetable Preservation Research Station, Campden, of obtaining high vacua in cans which can be used to supplement or replace the normal heat exhaust method. The cans are passed through a steam tunnel about 2 foot long which leads directly to the seaming head of the closing machine where a jet of steam plays across the surface of the contents whilst the can is being closed. A check after 1 year's storage showed that cans closed after steam injection have a vacuum 3″ higher than similar cans closed the normal way, i.e. after a heat exhaust of some 6 minutes at 190°F. It is claimed that the shelf life of such a can is some 6-30% longer.

Sommaire: Bref rapport concernant une nouvelle méthode d'évacuation de boîtes en fer blanc qui serait une amélioration par rapport à la méthode de séchage à chaud.

Note by Anon. *Food Manuf*, 1.1.1951 8

O

IV — Special Subsidiary Subjects — IV

Abstract No. and References

40 — GASES AND VAPOURS — 40

Construction of a New Combined Vacuum Furnace and Mass Spectrometer for Universal Use 41/IV

See Abstract No. : 105/II

Instrumentation (Several New Mass Spectrometers . . . .) 42/IV

See Abstract No. : 104/II

Low-Temperature Separation of Ethane from Methane and Air 43/IV

United States. The successful separation of ethane from methane and air is of importance, where geo-chemical methods are applied to the prospecting for oil. It is generally believed that the use of vacuum apparatus with liquid air or liquid nitrogen as the refrigerant is satisfactory for this purpose. In actual fact, however, the vapour pressure of ethane at — 196°C., the boiling point of liquid nitrogen at atmospheric pressure, is still high enough to cause some of the gas to be lost due to the action of the vacuum pumps. But, if the temperature at which the separation is effected, is lowered to —210°C., then the vapour pressure of ethane is reduced to $5.3 \times 10^{-6}\mu$ and the danger of losing ethane gas in the manner described is overcome. The author points out that the temperature of liquid nitrogen can be reduced to that value, if the container holding it is partially evacuated. He describes a freezing trap which is designed to hold the liquid nitrogen acting as the refrigerant in a Dewar flask evacuated to a pressure of 25—27 inches Hg. This trap, is incorporated in the vacuum system used for the analysis. Further details are given of the method employed to arrive at a good quantitative separation.

Article by R. L. Slobod *Analyt. Chem.* **23**, Feb. 1951 361-363

Sommaire : Description d'une méthode permettant la détermination précise de la teneur en éthane de mélanges air-méthane-éthane.

The Effect of Crystal Parameter on Hydrogenation and Dehydrogenation 44/IV

See Abstract No. : 100/III.

41 — FREEZE DRYING — 41

Further Investigations on the Preservation of Mold Cultures 45/IV

United States. Due to the extensive research work now carried out on antibiotic substances, lasting preservation of mold cultures has become a problem of great importance to the scientist. The authors report on their experiences with freeze-dried cultures of various materials over observation periods of up to 7 years. The table reproduced below in abbreviated form, summarizes the results of their observations :—

Viability of Freeze-Dried Preparations of Selected Mold Cultures Tested at Intervals up to 85 Months

Name	*NRRL No.*	*Test No. 1*		*Test No. 2*		*Test No. 4*		*Test No. 6*	
		Age in Months	*Viability*	*Age in Months*	*Viability*	*Age in Months*	*Viability*	*Age in Months*	*Viability*
Aspergillus flavus Link	693	2	Good	24½	Good	38½	Good	83½	Excellent
A. itaconicus Kinoshita	161	3½	Fair	26	Fair	41	Poor	84½	Fair
A. niger van Tieghem ...	3	4	Good	26	Good	41	Good	85	Excellent
A. niger van Tieghem ...	328	3	Excellent	25	Excellent	39½	Excellent	84	Excellent
Penicillium chrysogenum Thom	811	3½	Excellent	26	Excellent	40½	Excellent	85	Excellent
P. vinaceum Gilman and Abbott ...	739	2½	Fair	24½	Poor	38½	Poor	60	Poor

Three methods of preservation were used to arrive—by comparison—at a proper assessment of the quality of the freeze-drying method :—(1) preservation in plain agar slants, (2) preservation in fertile garden soil and (3)

Vol. 1 1951

IV — Special Subsidiary Subjects — IV

Contd.

Abstract No. and References

preservation under a mineral oil seal. Mucorales were chosen as test organisms. (1) Represents the conventional procedure practiced in most laboratories. (2) Is an adaptation of a technique established for some time now and applied to bacteria cultures, in particular, cultures of the anaerobic spore-forming clostridia. (3) is a method originally proposed by Ungermann in 1918 and extensively used by Sherf in 1943 for the maintenance of fungus cultures. The authors came to the conclusion that preservation in freeze-dried form is superior to preservation in any other form. In the case of Mucorale cultures, the techniques (2), (3) and (1) represent progressively less favourable methods.

Sommaire : Etude systématique des performances en tant que conservation de cultures fongueuses lyophilisées comprenant une période de 7 ans.

Article by D. I. Fennell K. B. Raper & M. H. Flickinger *Mycologia* **42**, Jan.-Feb. 1950 135-147

A Simplified Method of Lyophilizing Micro-organisms 46/IV

United States. A method of freeze-drying micro-organisms on a laboratory scale is described which requires a minimum of special equipment and materials. A piece of pyrex tubing is used, 8 mm. wide and 12 inches long, closed at the bottom. The sample to be freeze-dried is introduced by a pipette and a cotton plug is pushed down the tube to about 3½ inches above the sample. Then, a small amount of powdered phosphoric anhydride is placed on the cotton plug to act as a desiccant and held in position with a second cotton plug. A number of tubes prepared in this manner are immersed into a solvent-dry ice mixture for rapid freezing. At this stage each tube is individually connected to a rotary pump, evacuated and sealed off at the upper end. Subsequently it is placed into a freezing mixture consisting of glycol and water held at a temperature of -- 5 to — 10°C. After about 4 hours the sample is completely dry and the tube is finally sealed at a distance of some 2 inches above the sample. Comparing the method described here with others, it is claimed that the short distance between the sample and the desiccant assists in speeding-up desiccation.

Sommaire : Description d'un simple dispositif de laboratoire, de lyophylisation de matériel bactériologique.

Letter by R. W. Barratt & E. L. Tatum *Science*, **112**, 28.7.1950 122-123

45 — ELECTRONICS — 45

A Geiger-Counter Mounting for Continuous Counting of Gas-Phase Radioactivity 47/IV

See Abstract No. : 78/I

Experiments with an Electron Gun Designed for Use in Electron Diffraction Cameras 48/IV

France. The author discusses a new and simple design of an electron gun of the converging-beam type. At the same time he reports on a study covering a variety of electron gun designs and arrives at conclusions of great practical interest.

Sommaire : Description d'un nouveau canon à électrons du type à rayon convergent.

Article by P. Renaud *J. Phys. Radium* **3**, Nov. 1950 619-621

Metal-Backed Television Picture Tubes 49/IV

United States. Improved contrast and brightness of the picture are the two main items of research carried out in the field of television engineering all over the world. One result of this work is the provision of a thin metal coating (usually aluminium) on the bombarded side of the phosphorous screen in the tube. Brief reference is made to the method of producing these films by deposition *in vacuo*. Without such a metal backing the phosphorous screen is throwing no more than 50% of the light generated toward the face of the tube and of this amount some 20% are lost due to internal reflections in the glass envelope. As a result of the mirror finish of the metal coating, the amount of light thrown forward is almost doubled. This has the further advantage that the anode current can be reduced to half the value required for non-metallised screens, where the brightness of the picture produced without the provision of the film is satisfactory. Reducing the anode current means reducing the diameter of the electron beam, as a result of which the resolution quality of the picture improves or, in other words, its contrast. There are other advantages connected with the use of a metal backing. For instance, it prevents the formation of ion spot blemishes on the tube face without the use of ion trap guns and counteracts the adverse effects of voltage saturation of the screen material.

Sommaire : Discussion des caractéristiques et propriétés d'écrans de télévision phosphorescent, à face antérieure aluminisée produite par l'évaporation sous vide.

Article by C. T. Waugh *Telev. Engng.* **1**, Dec. 1950 12-14

The Physical Basis of the Residual Vacuum Characteristics of the Thermionic Valve 50/IV

United Kingdom. The experimental work described here is related to an investigation published by the author previously (see abstract No. 14/**IV**). Examining the residue factor characteristics of a number of valves, he found in all instances a marked discontinuity in the anode voltage range of 80 to 100. A literature search did not produce a satisfactory explanation of this phenomenon and therefore, it was decided to carry out experiments following up Richardson's work on reverse-grid-current effects in valves. Details and a sketch are

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

given of the experimental set-up. Means were provided for filtering out the radiation effects leaving the positive ions for direct measurement. All measurements were taken with the tube evacuated to a constant k_0 condition. No more than 3% of the total current measured could be accounted for by the flow of positive ions, i.e. almost the whole of the reverse-grid-current measured was due to x-radiation. If the assumptions, on which the use of the suppressor techniques applied in these experiments are based, can be verified, similar techniques could be employed in ionization gauges with the probable result that there is no longer a lower limit to accurate pressure measurement.

Sommaire : Des recherches sur les fondements physiques des caractéristiques de vide résiduel d'un tube thermionique conduit à des résultats ayant de l'importance pour la construction des jauges à ionisation.

Article by G. H. Metson *Brit. J. Appl. Phys.* **2**, Feb. 1951 46-48

The Spectral Emittance of Nickel- and Oxide-Coated Nickel Cathodes.

51/IV

United Kingdom. Little information has been published on the spectral emittance of cathodes used in thermionic valves and some of the values quoted differ widely. The work of Champeix is mentioned who dealt with the influence of coating thickness and temperature on the spectral emittance. The authors of the present article investigated other factors which could influence the true emittance or the experimental determination of emittance of bare nickel cathodes or nickel cathodes coated with barium strontium oxide. In the case of the bare nickel cathode these factors were :—the variation of emittance due to stoving treatment and the effect of getters used in the tube. In the case of the oxide-coated cathode they included :—the effects of degassing the glass envelope, of contamination by gettering materials and changes in surface texture of the coating. For bare nickel cathodes, it was found that in all cases emittance changed with temperature by a constant value in the range from room temperature to 800°C. In higher temperature regions a marked rise was noted. The average temperature coefficient was 2.4×10^{-4} deg^{-1}. The emittance value of coated cathodes assembled to tubes baked below 400°C. and gettered with non-evaporating zirconium averaged 0.15. Using a magnesium barium getter increased this value to 0.24. Coating composition, texture of surface and method of coating affected the emittance appreciably.

Sommaire : Présentation des résultats d'expérience concernant l'émission (émitance) spectrale de cathodes utilisées dans des tubes thermioniques.

Article by S. L. Martin & G. F. Weston *Brit. J. Appl. Phys.*, **1**, Dec. 1950 318-324

Vol. 1 1951

47 — GLASS PROCESSING — 47

Nickel-Chromium-Iron Alloy for Sealing to Glass

52/IV

United Kingdom. Some years ago a special alloy was developed in the United States for sealing to soft lead glass of the type used in the manufacture of lamps and valves. Its composition was :—nickel = 42%, chromium = 6% and iron = 52%. Its main virtue was believed to lie in the fact that its Curie temperature and glass transformation temperature as much as the expansion coefficient of both materials above and below this temperature were almost the same. In the view of the author, however, it is less important that the values just quoted are almost identical as long as they are related in a manner which ensures sufficiently low a stress at any temperature. The alloy of the composition given above did not fulfil this condition. Seals of that type showed no stress at room temperature but appreciable stress in the temperature region of about 300°C. It appeared that a higher nickel content was desirable. Nine materials of different composition were tested with the result that an alloy containing 47% nickel and 5% chromium was proved to give an excellent match showing very low stress at all values of the temperature range considered in spite of the fact that the alloy Curie temperature was appr. 340°C. and the glass transformation temperature about 400°C.

Sommaire : L'étude méthodique des facteurs régissant un bon joint métal-verre (mou) aboutit au développement d'un nouvel alliage nickel-chrome-fer particuliérement convenable à ce but.

Article by J. E. Stanworth *J. Sci. Instrum.* **27**, Oct. 1950 282-284

Cumulative Author Index

(For Abstract Nos. 1/I — 19/I; 1/II — 43/II; 1/III — 48/III; 1/IV — 22/IV see January Issue)
(For Abstract Nos. 20/I — 54/I; 44/II — 83/II; 49/III — 88/III ; 23/IV — 40/IV see April Issue)

Author	Title of Publication	Abstr. No.
Affleck, J. H., et al.	The Barium Oxide on Tungsten Cathode Interfaces.	36/IV
Agar, A. W., et al.	Replica Techniques in Electron Microscopy.	102/II
Aitchison, P. M., et al.	Two Improved Methods of Graticule Production Using Evaporated Metal Films.	65/III
Aitchison, R. E., et al.	Two Improved Methods of Graticule Production Using Evaporated Metal Films.	65/III
Alberman, K. B.	A Small High-Temperature High-Vacuum Furnace.	118/III
Aldington, J. N.	Some Recent Developments in Rare-Gas Discharge Lamps.	36/I
Alexander, P., et al.	Copper-Titanium Coating and Bonding Process.	75/III
Alexander, P.	Improvements i.o.r.t. Methods of Eliminating Moisture from the Surfaces of Moisture Absorbant Sheet Material on Which Metal is to be Deposited By Thermal Evaporation in a Vacuum.	24/III
Allen, J. A., et al.	The Adsorption of Gases on Copper Films.	58/III
Allison, H. W., et al.	Thermionic Emission of Thin Films of Alkaline Earth Deposited by Evaporation.	15/IV
Alpert, D., et al.	Extension of the Low Pressure Range of the Ionization Gauge.	57/II
Andrade, E. N. da C., et al.	The Thermal Etching of Single Crystals of Cadmium.	12/I
Andrews, A. I., et al.	A Quenching Furnace for High Temperature Studies of Small Specimens.	117/III
Andrews, E. B., et al.	Changes in the Reactivity of Purified Prothrombin After Freeze-Drying.	33/IV
Anon.	The Electronic Telescope.	26/II
Anon.	Porcelain, Its Manufacture and Uses.	33/II
Anon.	Gilding the Lily.	10/III
Anon.	Seeing Molecules.	11/III
Anon.	Uranium in Microscopy.	12/III
Anon.	Cathodic Vacuum Etching Process.	23/III
Anon.	Distillation Process for Fats.	39/III
Anon. (R.L.K.)	Defatting Distillation.	40/III
Anon.	Multi-Column Reflux Still.	41/III
Anon.	Cream Fondant by Vacuum Methods.	45/III
Anon.	Vacuum Processing Apples Prevents Browning.	46/III
Anon.	Vacuum Cooling.	48/III
Anon.	Diaphragm Micromanometer with Electrical Pick Up.	55/II
Anon.	The Field Emission Electron Microscope Makes Molecules Visible.	64/II
Anon.	R. F. Field Mass Spectrometer.	69/II
Anon.	Double Distillation.	68/III
Anon.	A Hardness Tester for Thin Films.	90/III
Anon.	Vacuum Metallurgy.	115/III
Anon.	H.F. Induction Furnaces.	116/III
Anon.	Chromium Salvage.	119/III
Anon.	Analytical Distillation.	122/III
Anon.	A Low-Cost Still Removes Light Ends from Fatty Acids.	129/III
Anon.	High Vacua in Cans.	133/III
Arneil, A., et al.	De-Odorization of Vegetable Oils.	44/III
Ashmead, J.	A Joule-Thomson Cascade Liquefier for Helium.	81/II
Atkins, C. D., et al.	New Evaporator Robotized for High Output Efficiency.	70/III
Atkins, W. F., et al.	The Design of a Large Vacuum Valve for Application to an Electric Furnace.	35/II
Axelbank, M.	A Rotary McLeod Gauge.	7/II
Backus, R. C., et al.	The Use of Spraying Methods and of Volatile Suspending Media in the Preparation of Specimens for Electron Microscopy.	20/II
Bacot, P. A., et al.	Process of Preparing Coffee Extract.	132/III
Bacquet, M. E. H.	The Practice of Cathodic Sputtering.	66/III
Barratt, R. W., et al.	A Simplified Method of Lyophilizing Micro-organisms.	46/IV
Bas-Taymaz, E.	The Service Life of Glowing Tungsten Filaments and Their Speed of Evaporation in High Vacuum.	31/I
Bashkin, S. et al.	Ion Gauges.	95/II
Batchelder, A. H., et al.	Biological Window.	79/I
Bayard, R. T., et al.	Extension of the Low Pressure Range of the Ionization Gauge.	57/II
Beach, A. L., et al.	A Vacuum Fusion Furnace for the Analysis of Gases in Metals.	31/III
Beams, J. W., et al.	Magnetically Suspended Vacuum-Type Ultra-centrifuge.	108/II
Beeck, O., et al.	Determination of Heats of Adsorption Using Metal Films.	59/III
Beeck, O., et al.	The Effect of Crystal Parameter on Hydrogenation and Dehydrogenation.	100/III
Benner, F. C., et al.	Remote-Condenser Falling-Film High Vacuum Still.	84/III
Benson, N. C., et al.	A 72" High Vacuum Evaporator and its Glow Discharge Equipment.	107/III
Biguenet, Ch.	The Photo-Adsorption of a Thin Film of Barium.	37/IV

Cumulative Author Index

Contd.

(For Abstract Nos. 1/I — 19/I; 1/II — 43/II; 1/III — 48/III; 1/IV — 22/IV see January Issue)
(For Abstract Nos. 20/I — 54/I; 44/II — 83/II; 49/III — 88/III; 23/IV — 40/IV see April Issue)

Author	Title of Publication	Abstr. No.
Birks, L. S., et al.	Particle Size of Evaporated Gold.	15/III
Blackburn, P. E., et al.	Vapour Pressure of Inorganic Substances IV. Tantalum between 2,624 and 2,493°K.	73/I
Blasco, E., et al.	A New Pirani-Type Vacuum Gauge.	10/II
Blewett, J. P., et al.	Design Study for a Three-Bev Proton Accelerator.	16/IV
Blom, E C.	A New Method of Measuring Pressure Electrically.	53/II
Bockris, J. O'M., et al.	A Vacuum Furnace for Use in the Temperature Range of 1,000°-2,000°C.	32/III
Boettcher, A.	On the Production and Structure of Layers Condensed in Vacuo and Consisting of Aluminium Silver and Aluminium Magnesium Alloys.	97/III
Bonner, F., et al.	A Geiger-Counter Mounting for Continuous Counting of Gas-Phase Radioactivity.	78/I
Boorse, H. A., et al.	Design and Performance of a Joule-Kelvin Helium Liquefier.	120/II
Booth, P. R.	Device for Use with Water-Driven Vacuum Pumps to Prevent Water Sucking back into the Apparatus.	116/II
Bowen, R. J., et al.	Dry Metallic Friction as a Function of Temperature between 4.2°K and 600°K.	68/I
Boyd, C. A., et al.	A Method of Making Glass-To-Metal Seals.	22/IV
Bradbury, S., et al.	Process for the Desiccation of Acqueous Materials From the Frozen State.	34/IV
Breger, I. A., et al.	Vacuum Differential Thermal Analysis.	87/III
British Aluminium Co. Ltd., et al.	Improvements in the Manufacture of Metallic Titanium.	76/III
British-Thomson-Houston Co. Ltd.	Improvements i.o.r.t. Non-Reflecting Optical Bodies.	3/III
Brockmyre, H. F., et al.	Mass Spectrometric Analysis of Low Concentrations of Vapours in Air.	26/IV
Brown, J.	Modern Practice in Investment Casting.	26/III
Brown Boveri & Co., A. G	Device for Making a Vacuum-Tight Connection Between Two Metallic Parts of Different Electric Potential.	21/IV
Bull, T. H., et al.	Life-Time of Potassium Ions on a Tungsten Filament at 1,460°K.	77/I
Burmaster, K. E.	Experimental High Megohm Resistors.	8/I
Burmaster, K. E., et al.	A Philips Type Ionization Gauge for Measuring of Vacuum from 10^{-7} to 10^{-1}mm. Hg.	56/II
Butler, C. P., et al.	A Motor for Use in Vacuum Systems.	37/II
California Research Corp., et al.	Biological Window	79/I
Carpenter, F. E., et al.	A Motor for Use in Vacuum Systems.	37/II
Carpenter, L. G., et al.	The Evaporation of Titanium.	123/II
Chambers, M. A., et al.	An Investigation of High Vacuum Freeze Drying as a Means of Drug Preservation—Part I.	32/IV
Champeix, R.	On the Possibility of Using the Ionization of Gas Molecules for the Production of Low Pressures.	45/II
Clancey, V. J.	Evaporation and Mobility of Naphthalene Molecules.	76/I
Clark, J. W., et al.	An Automatic Control of High Vacuum Systems.	39/II
Clark, R. K.	The Absolute Determination of the Emission Rate of Beta-Rays.	39/I
Clark, W. J.	Tele-recording of Thickness, Pressure Flow & Other Physical Quantities Using a Simple Electromagnetic Circuit.	9/II
Cloud, R. W., et al.	Vacuum Tests of Rubber, Lead and Teflon Gaskets and Vinyl Acetate Joints.	71/II
Coggeshall, N. D., et al.	A Portable Gas Blending System.	4/IV
Cole, W. A., et al.	Determination of Heats of Adsorption Using Metal Films.	59/III
Conner, J. P., et al.	Preparation of Thin Tritium Targets.	130/III
Consolidated Engineering Corp., et al.	Method for Treating Filaments of Ionization Gauges.	58/II
Corran, H. S.	Technical Developments of Fermentation Processes.	12/IV
Coslett, V. E.	Preparation of Specimens for the Electron Microscope.	8/III
Coulson, J. M., et al.	Manufacture of Mononitrotoluene.	37/III
Courtney-Pratt, J. S.	Direct Optical Measurements of the Length of Organic Molecules.	14/I
Coyle, R. A., et al.	A Multiple High-Vacuum Valve Unit.	115/II
Croft, A. J., et al.	Method of Handling & Storing Liquefied Gases.	7/IV
Dart, F. E.	Evaporation of Zinc and Zinc Oxides in Electron Bombardment.	41/I
Dash, J. G., et al.	Design and Performance of a Joule-Kelvin Helium Liquefier.	120/II
Davis, W., et al.	A System for the Collection and Purification of Radon by Remote Control.	83/II
Dekker, A. J., et al.	Hall Coefficient and Resistivity of Thin Films of Antimony Prepared by Distillation.	56/III

Vol. 1 1951

Cumulative Author Index

Contd.

(For Abstract Nos. 1/I — 19/I; 1/II — 43/II; 1/III — 48/III; 1/IV — 22/IV see January Issue)
(For Abstract Nos. 20/I — 54/I; 44/II — 83/II; 49/III — 88/III; 23/IV — 40/IV see April Issue

Author	Title of Publication	Abstr. No.
Derge, G.	Analysis of Oxygen in Titanium.	30/III
Descamps, C., et al.	Calculation of Interference Filters.	48/I
Deutsch, Z. G., et al.	Mass Spectrometer for Leak Detection.	41/II
Dillon, J. F., et al.	Magnetically Suspended Vacuum-Type Ultra-Centrifuge.	108/II
Dinardo, A., et al.	Remote-Condenser Falling-Film High Vacuum Still.	84/III
Distillation Products Inc.	Improvements i.o.r.t. the Heating or Rotating Workpieces by Electromagnetic Induction.	128/III
Dixon, O. G.	High-Efficiency Laboratory Fractionation. IV. A Metal Laboratory-Still.	85/III
Dodd, C.	The Electrical Resistance of Liquid Gallium in the Neighbourhood of Its Melting Point.	30/I
Doelling, R. F., et al.	Vacuum Treatment of Parkes' Process Crusts on a Pilot-Plant Scale.	120/III
Dornfield, E. G., et al.	A Small Electron Microscope.	99/II
Duckworth, J. E.	A Large Dempster-Type Double-Focusing Mass Spectrograph.	27/II
Dufour, C.	Evaporation of Thin Films of Uniform and Controlled Thickness. Their Application to Electronics.	52/III
Dunoyer, L.	Comments on Formulae for the Flow of Gases in Pipes at Low Pressures.	24/IV
Dunoyer, L.	The Action of a Gas on a Solid.	28/IV
Dunoyer, L.	On Certain Phenomena Concerning the Release of Gases Observed in the Production of Low-Voltage Incandescent Lamps.	6/I
Eckstein, N. A., et al.	A Method of Making Glass-To-Metal Seals.	22/IV
Edwards, J. W., et al.	Vapour Pressure of Inorganic Substances IV. Tantalum between 2,624 and 2,493°K.	73/I
Edwards, J. W., et al.	Vapour Pressure of Inorganic Substances V. Zirconium between 1,949 and 2,054°K.	74/I
W. Edwards & Co. (London) Ltd., et al.	Improvements in Measuring Devices Dependent on Radiometer Action.	6/II
Eisner, E.	Phase Change of Light on Reflection at a Mica-Silver Interface.	96/III
Ellis, O. W., et al.	A Horizontal High Vacuum Electrical Resistance Furnace.	79/III
Elmes, A. W., et al.	Improvements Relating to Enamelling.	43/III
Engel, A. von., et al.	Development of the Low Pressure Electrodeless Discharge in a High Frequency Electric Field.	35/I
Espe, W., et al.	Getter Materials for Electron Tubes.	24/I
Espe, W.	Plated Iron as a New Material in Vacuum Technique.	109/II
Evans, E. C., et al.	A Philips Type Ionization Gauge for Measuring of Vacuum from 10^{-7} to 10^{-1}mm. Hg.	56/II
Ewald, E. R.	Manufacturing Metallised Picture Tubes.	7/III
Fast, J. D.	Apparatus for the Preparation of Metals with an Exactly Known Content of Impurities.	33/III
Fehlberg, E., et al.	New Evaporator Robotized for High Output Efficiency.	70/III
Fein, M. L., et al.	An Improved Manostat and Manometer.	40/II
Feller, M., et al.	Determination of Liquid-Vapour Equilibria.	3/IV
Fennell, D. I., et al.	Further Investigations on the Preservation of Mold Cultures.	45/IV
Ferris, S. W., et al.	Guided Free-Film Distillation Apparatus.	82/III
Filosofo, I., et al.	Measuring Low Vapour Pressures.	79/II
Fischbein, I. W.	Electron Microscopy of Wet Biological Tissues by Replica Techniques.	101/II
Fisher, J. H.	Metallised Paper Capacitors.	28/I
Fitzgerald, J. W., et al.	A Method of Making Glass-To-Metal Seals.	22/IV
Flickinger, M. H., et al.	Further Investigations on the Preservation of Mold Cultures.	45/IV
Fochs, P. D.	A Method of Determining Concurrently the Thickness and Refractive Index of a Thin Film or Lamina.	45/I
Food Technology Inc.	Coconut Processing.	88/III
Fortune, W. B., et al.	Antibiotics Development.	25/III
Francis, G., et al.	Development of the Low Pressure Electrodeless Discharge in a High Frequency Electric Field.	35/I
Franzen, W., et al.	A Reliable Pirani Vacuum Safety Circuit.	121/II
Fulkerson, S. D., et al.	Apparatus for Determining Gas Permeability of Carbon and Graphite.	80/II
Garfitt, R. G., et al.	A Three-Stage Electron Microscope with Stereographic Dark Field and Electron Diffraction Capabilities.	19/II
Garrod, R. I.	A Low-Impedance High Vacuum Valve.	36/II
Garrod, R. I., et al.	A Multiple High-Vacuum Valve Unit.	115/II
Gauer, O. H., et al.	A Miniature Pressure-Recording Device.	91/II

Cumulative Author Index

Contd.

(For Abstract Nos. 1/I — 19/I; 1/II — 43/II; 1/III — 48/III; 1/IV — 22/IV see January Issue)
(For Abstract Nos. 20/I — 54/I; 44/II — 83/II; 49/III — 88/III ; 23/IV — 40/IV see April Issue)

Author	Title of Publication	Abstr. No.
Geach, G. A., et al.	A Laboratory Arc-Furnace for Melting Alloys Containing the Refractory Transition Metals.	78/III
Geoghegan, D.	A Simple Trap for Use with a Water Pump	117/II
Gibson, A. F., et al	The Photo-Conductivity of Bismuth Sulphide and Bismuth Telluride.	16/III
Gienapp, E., et al.	Miniature Pressure-Recording Device.	91/II
Gilmont, R.	Design and Operational Characteristics of Cartesian Manostats.	122/II
Gilson, A. R.	Short-Path Vacuum Stills.	127/III
Ginsborg, B. L., et al.	The Deposition of Rhodium on Perspex.	105/III
Glabe, E. F.	Coconut Processing.	88/III
Glass, H. M., et al.	Improvements Relating to Enamelling.	43/III
Glober, J. A., et al.	Glass Circulating Pump.	2/II
Goldschwartz, J. M.	A High-Speed Ionisation Gauge.	94/II
Green, G. K., et al.	Design Study for a Three-Bev Proton Accelerator.	16/IV
Green, J., et al.	The Concentration of Tocopherols from Natural Sources by Molecular Distillation.	81/III
Greenwood, H. W.	Infiltrated or Impregnated Alloys.	54/I
Grinrod, J.	A Swedish Method of Wood Preservation.	67/III
Groszkowski, J.	A McLeod Gauge of Multiple Compression.	8/II
Grouse, R. A., et al.	Improvements i.o.r.t. Metallised Paper Capacitors.	62/III
Guldner, W. G.	Determination of Carbon in Ferrous Alloys.	29/III
Guldner, W. G., et al.	A Vacuum Fusion Furnace for the Analysis of Gases in Metals.	31/III
Haase, G.	The Gettering Efficiency of Thin Barium Films at Low Pressures.	25/I
Haine, M. E., et al.	A Three-Stage Electron Microscope with Stereographic Dark Field, and Electron Diffraction Capabilities.	19/II
Hall, C. E.	A Low Temperature Replica Method for Electron Microscopy.	9/III
Halsted, R. E., et al.	Gas Flow Through the Mass Spectrometer Viscous Leak.	106/II
Hamm, F. A.	Electron Microscope.	17/II
Hamm, F. A.	Symposium on Thin Films.	2/III
Hanna, J. G., et al.	Direct Determination of Oxygen in Compounds Containing Carbon, Hydrogen and Oxygen.	51/I
Happ, G. P., et al.	Mass Spectrometric Analysis of Low Concentrations of Vapours in Air.	26/IV
Happel, W. J., et al.	A Portable Gas Blending System.	4/IV
Harris, J. E., et al.	New Techniques in Auto-Radiography.	10/IV
Hass, G., et al.	A 72" High Vacuum Evaporator and its Glow Discharge Equipment.	107/III
Havens, R., et al.	A New Vacuum Gauge.	54/II
Haworth, L. H., et al.	Design Study for a Three-Bev Proton Accelerator.	16/IV
Heavens, O. S., et al.	The Deposition of Rhodium on Perspex.	105/III
Heid, J. L.,	Frozen Concentrated Citrus Juice Process Relies on Instruments.	114/III
Heller, R. B.	Cutting Flat Vacuum Gaskets on a Lathe.	38/II
Helwig, G., et al.	The Structure of Obliquely Evaporated Films and Their Influence on the Formation of Sub-Microscopic Surface Irregularities.	13/III
Henry, R.	Measurement of the Pumping Speed of Rotary Pumps.	50/II
Hensley, E. B., et al.	The Barium Oxide on Tungsten Cathode Interfaces.	36/IV
Hercus, G. R., et al.	A Medium Capacity Helium Liquefier.	119/II
Hibi, T., et al.	On the Method of Determining the Best Operating Condition of a Fractionating Oil Diffusion Pump.	84/II
Hickman, K. C. D.	Reverse Separation in Fractionation Pumps.	85/II
Hickman, K. C. D.	High Vacuum Distillation.	36/III
Hickman, K. C. D.	High Vacuum Distillation.	124/III
Hicks, T. J.	How To Figure Reciprocating Vacuum Pumps.	1/II
Hipple, J. A., et al.	Mass Spectrometry.	16/II
Hoene, J. von, et al.	Determination of Oxygen in Metal by the Vacuum Fusion Method.	123/III
Honig, R. E.	A Greaseless Gas Flow Valve.	59/I
Hopfield, J. J.	Glass Variable Microleaks for Gases.	76/II
Hopke, E. R., et al.	An Effective Vacuum Cut-Off.	75/II
Hopkins, E. G.	Vacuum Tubes with Mutually Bombarding Oxide Cathodes.	38/IV
Horton, J., et al.	A Reliable Pirani Vacuum Safety Circuit.	121/II
Hunt, Ltd., A. H., et al.	Improvement i.o.r.t. Metallised Paper Capacitors.	62/III
Hurd Corp., et al.	Apparatus and Method for Evaporation and Concentration of Liquids.	69/III
Ilfeld, R. M.	A Recording Mass Spectrometer.	28/II
Imperial Chemical Industries, et al.	Improvements Relating to Enamelling.	43/III
Isikawa, K., et al.	On the Method of Determining the Best Operating Condition of a Fractionating Oil Diffusion Pump.	84/II

Cumulative Author Index

Contd.

(For Abstract Nos. 1/I — 19/I; 1/II — 43/II; 1/III — 48/III; 1/IV — 22/IV see January Issue)
(For Abstract Nos. 20/I — 54/I; 44/II — 83/II; 49/III — 88/III; 23/IV — 40/IV see April Issue)

Author	Title of Publication	Abstr. No.
Jacobs, R. B.	The Design of Molecular Pumps.	87/II
Jacobs, R. B., et al.	Improvements i.o.r.t. the Heating of Rotating Workpieces by Electromagnetic Induction.	128/III
Jenkins, G. C. H., et al.	The Design of a Large Vacuum Valve for Application to an Electric Furnace.	35/II
Johnson, Jr., E. F.	Evacuation Time with Reciprocating Pumps	44/II
Johnson, F. C., et al.	Glass Circulating Pump.	2/II
Johnson, G. W.	Surface Tension Effects on Oriented Thin Silver Films.	14/III
Johnson, P. D.	Behaviour of Refractory Oxides of Metals Alone and in Combination in Vacuo at High Temperatures.	34/II
Johnston, H. L., et al.	Vapour Pressure of Inorganic Substances IV. Tantalum between 2,624 and 2,493°K.	73/I
Johnston, H. L., et al.	Vapour Pressure of Inorganic Substances V. Zirconium between 1,949 and 2,054°K.	74/I
Jolly, W. P., et al.	The Pirani Effect in a Thermionic Filament as a Means of Measuring Low Pressures.	4/II
Jones, D. O., et al.	Method of Handling and Storing Liquefied Gases.	7/IV
Keeping, Jr., G. R.	On the Growth of Metallic Crystals from the Vapour Phase.	5/III
Kenty, C.	McLeod Gauge with an Electrical Contact.	92/II
Kern, R. A., et al.	The Apparent Size of Objects as Observed in the Electron Microscope.	32/II
Kern, S. F., et al.	The Apparent Size of Objects as Observed in the Electron Microscope.	32/II
Kerner, H.	Construction of an All-Glass Mercury Circulation Pump.	88/II
Kervenski, I. R., et al.	Direct Determination of Oxygen in Compounds Containing Carbon, Hydrogen and Oxygen.	51/I
Kiberlin, C. N., et al.	Lubricants Produced by Reactions in Glow Discharge.	131/III
King, T. P., et al.	New Techniques in Auto-Radiography.	10/IV
Kirby, D.	Pure Oxide Refractories.	113/II
Knoll, M., et al.	Getter Materials for Electron Tubes.	24/I
Knox, F. A., et al.	Vacuum Pumping Method with Mechanical Oil Sealed Pumps.	89/II
Koenig, H., et al.	The Structure of Obliquely Evaporated Films and Their Influence on the Formation of Sub-Microscopic Surface Irregularities.	13/III
Koll, R., et al.	A New Vacuum Gauge.	54/II
Kroll, W. J.	Improvements in Processes and Apparatus for the Treatment of Hafnium, Zirconium, Titanium, Thorium and Their Alloys, in the Molten State.	27/III
Kroll, W. J., et al.	Production of Malleable Zirconium.	35/III
Kuhn, H., et al.	Reflectivity of Thin Silver Films and Their Use in Interferometry.	55/III
Kuhn, W. E., et al.	A Horizontal High Vacuum Electrical Resistance Furnace.	79/III
Lagow, H., et al.	A New Vacuum Gauge.	54/II
Lambert, F. J., et al.	Apparatus for Determining Gas Permeability of Carbon and Graphite.	80/II
Lampitt, L. H.	Chemistry in Food Preservation.	15/I
Lamson, E. R., et al.	Guided Free-Film Distillation Apparatus.	82/III
Laporte, M.	Lamps Giving a White Light of Great Intensity.	67/I
Leduc, P.	Precautions to be Taken in the Use of the Philips Gauge.	15/II
Lesh, J. B., et al.	Fractionation of Blood Plasma.	9/IV
Leverton, W. F., et al.	Hall Coefficient and Resistivity of Thin Films of Antimony Prepared by Distillation.	56/III
Lillie, A. B., et al.	Preparation of Thin Tritium Targets.	130/III
Livingston, M. S., et al.	Design Study for a Three-Bev Proton Accelerator.	16/IV
Lloyd, J. T.	An Audible Vacuum Leak Detector.	42/II
Lomas, J.	Plating by Vacuum Evaporation.	89/III
Lomer, P. D.	The Dielectric Strength of Aluminium Oxide Films.	57/III
Longini, R. L.	Evaporation and Outgassing in an Inert Atmosphere.	11/I
Lorant, M.	Helium Disappearance in Arc Discharge Tubes.	20/IV
Lyophile Cryochem. Corp., et al.	Process for the Desiccation of Acqueous Materials from the Frozen State.	34/IV
Mair, W. N., et al.	The Evaporation of Titanium.	123/II
Male, D.	A Graphical Method of Determining Simultaneously Optical Constants and the Thickness of Thin Metal Layers.	69/I
Marshall, D. G., et al.	Life-Time of Potassium Ions on a Tungsten Filament at 1,460°K.	77/I
Martin, S. L.	Vacuum Technique.	55/I
Martin, S. L. et al.	The Spectral Emittance of Nickel- and Oxide-Coated Nickel Cathodes.	51/IV
Masch, L. W.	Molecular Distillation in Production and in the Laboratory.	80/III
Matalon, R., et al.	A Simplified Design for a Molecular Still of the Horizontal Type.	38/III

Cumulative Author Index

Contd.

(For Abstract Nos. 1/I — 19/I; 1/II — 43/II; 1/III — 48/III; 1/IV — 22/IV see January Issue)
(For Abstract Nos. 20/I — 54/I; 44/II — 83/II; 49/III — 88/III; 23/IV — 40/IV see April Issue)

Author	Title of Publication	Abstr. No
Matheson, G. L., et al.	Lubricants Produced by Reactions in Glow Discharge.	131/III
McClaughry, R. I., et al.	Changes in the Reactivity of Purified Prothrombin After Freeze-Drying.	33/IV
McCormick, S. L., et al.	Antibiotics Development.	25/III
McCutcheon, D. M., et al.	Cathodic Vacuum Etching of Metals.	23/III
McDonald, H. J., et al.	Determination of Liquid-Vapour Equilibria.	3/IV
McFee, R. H.	A Method of Supplying Low-Pressure Gases to a Vacuum System.	5/I
McLauchlan, T. A., et al.	The Thickness Measurement of Thin Films by Multiple-Beam Interferometry.	51/III
McLauchlan, T. A., et al.	Continuous Observations with the Electron Microscope of the Formation of Evaporated Films of Silver, Gold and Tin.	98/III
Mclean, D. A.	Metallised Paper for Capacitors.	62/I
McMahon, H. O., et al.	Dry Metallic Friction as a Function of Temperature Between 4.2°K and 600°K.	68/I
Meer, S. van der, et al.	Automatic Regulation of Thermionic Emission.	17/IV
Menter, J. W., et al.	Apparatus for the Study by Electron Diffraction of the Effect of Temperature on Surface Films.	63/II
Merlin, M., et al.	Measuring Low Vapour Pressures	79/II
Merrison, A. W., et al.	The Harwell Time-of-Flight Neutron Spectrometer.	107/II
Merwe, J. H. van der., et al.	Low Reflectance Coatings and the Sensitivity Curve of the Eye.	72/I
Metal Hydrides Inc., et al.	Copper-Titanium Coating and Bonding Process.	75/III
Metcalfe, A. G.	The Use of Platinum Thermocouples in Vacuo at High Temperatures	66/I
Metson, G. H.,	Vacuum Factor of the Oxide Cathode Valve.	14/IV
Metson, G. H.	The Physical Basis of the Residual Vacuum Characteristic of a Thermionic Valve.	50/IV
Miller, G. L.	Zirconium.	77/III
Ministry of Supply	Improvements i.o.r.t. Pressure Measuring Apparatus.	93/II
Miranda, L., et al.	A New Pirani-Type Vacuum Gauge.	10/II
Mitchell, J. W., et al.	The Adsorption of Gases on Copper Films.	58/III
Mitchell, T. J.	The Rate of Evaporation in the Determination of Water.	85/I
Montes, L. M. Gonzalez, et al.	Industrial Applications of High Vacuum Techniques.	20/I
Moore, G. E., et al.	Thermionic Emission of Thin Films of Alkaline Earth Deposited by Evaporation.	15/IV
Morgan, Jr. W. E.	Fireproof Still.	42/III
Moss, T. S., et al.	The Photo-Conductivity of Bismuth Sulphide and Bismuth Telluride.	16/III
Moss, T. S.	The Ultimate Limits of Sensitivity of Lead Sulphide and Telluride Photo-Conductive Detectors.	44/I
Mukherjee, N. R., et-al.	Performance of a Vapour-Lift Pump under Vacuum.	46/II
Munch, R. H.	Instrumentation (Several New Mass Spectrometers . . .).	104/II
Myers, R. H.	Some Properties of Tantalum.	110/II
Nachod, F. C., et al.	Molecular Still.	126/III
Nash, L. K.	Gas Analysis.	1/IV
Nelson, J. W., et al.	An Investigation of High Vacuum Freeze-Drying as a Means of Drug Preservation.	32/IV
Nelson, L. S.	A Method of Determining the Thickness of Silver Deposited on Glass.	91/III
Nielson Jones, W., et al.	Preservation of Fungal Sporophores	11/IV
Nienhuis, K., et al.	Construction and Applications of a New Design of the Philips-Type Vacuum Gauge.	11/II
Nier, A. O., et al.	Gas Flow Through the Mass Spectrometer Viscous Leak.	106/II
Nineuil, P.	Electrolytic Polishing and Vacuum Engineering.	4/I
Oketani, S., et al.	An Apparatus for the Study of Insulating Surfaces by Means of Electron Diffraction.	62/II
Ortlieb, A.	Metallised Paper Recorder.	64/I
Oyama, S.	On the Vapour Flow in the Diffusion Pump.	48/II
Page, R. S., et al.	A Three-Stage Electron Microscope with Stereographic Dark Field and Electron Diffraction Capabilities.	19/II
Pahl, W., et al.	Cathodic Vacuum Etching of Metals.	23/III
Partridge, S. M.	Rotary Film Evaporator for Laboratory Use.	113/III
Patterson, D. C., et al.	Experimental Falling-Film Evaporator.	112/III
Penning, F. M., et al.	Construction and Applications of a New Design of the Philips-Type Vacuum Gauge.	11/II
Phillips, G. C., et al.	Preparation of Carbon Targets.	86/III

Vol. 1 1951

Cumulative Author Index

Contd.

(For Abstract Nos. 1/I — 19/I; 1/II — 43/II; 1/III — 48/III; 1/IV — 22/IV see January Issue)
(For Abstract Nos. 20/I — 54/I; 44/II — 83/II; 49/III — 88/III ; 23/IV — 40/IV see April Issue)

Author	Title of Publication	Abstr. No.
Phillips, M. A.	High Efficiency Vacuum Method. Simple and Effective Concentration of Aqueous Solutions.	111/III
Philp, S. E., et al.	Vacuum Tests of Rubber, Lead and Teflon Gaskets and Vinyl Acetate Joints.	71/II
Pierree, C., et al.	Checking the Purity of Hydrocarbon Liquids by Means of Electron Diffraction.	82/I
Plumlee, R. H., et al.	Mass Spectrometric Study of Solids. I—Preliminary Study of Sublimation Characteristics of Oxide Cathode Materials.	35/IV
Porsche, J. D., et al.	Fractionation of Blood Plasma.	9/IV
Powell, E. O., et al.	Detection & Measurement of Organic Vapours.	2/IV
Pratt, J. R. C., et al.	Performance of a Vapour-Lift Pump under Vacuum.	46/II
Preuss, L. E., et al.	Motion Picture Studies of Electron Bombardment of Colloidal Crystals.	65/II
Priznar, M., et al.	Molecular Still.	126/III
Raether, M. H.	Application of Electron Diffraction to the Study of Surface Films.	61/II
Raible, F., et al.	Mass Spectrometer for Leak Detection.	41/II
Randall, R. F. Y., et al.	The Thermal Etching of Single Crystals of Cadmium.	12/I
Raper, K. B., et al.	Further Investigations on the Preservation of Mold Cultures.	45/IV
Rasmussen, R. R., et al.	Vapour-Liquid Equilibria at Subatmospheric Pressures.	125/III
Ratchford, W. P., et al.	An Improved Manostat and Manometer.	40/II
Reis, D. van	An Air-Cooled High Vacuum Pump.	86/II
Reisner, J. H., et al.	A Small Electron Microscope.	99/II
Renaud, P.	Experiments with an Electron Gun Designed for Use in Electron Diffraction Cameras.	48/IV
Revell, R. S. M., et al.	Replica Techniques in Electron Microscopy.	102/II
Rhode-Hamel, H. W., et al.	Antiobiotics Development.	25/III
Richardson, J. E., et al.	Preparation of Carbon Targets.	86/III
Richardson, R. W., et al.	Lubricants Produced by Reactions in Glow Discharge.	131/III
Ritchie, A. W., et al.	The Effect of Crystal Parameter on Hydrogenation and Dehydrogenation.	100/III
Rogers, R. R., et al.	Refining Lithium by Vaporization at Low Pressure.	121/III
Roig, J., et al.	Calculation of Interference Filters.	48/I
Rose, W.	Packless Valve Flow Regulator.	74/II
Ross, J. D., et al.	Magnetically Suspended Vacuum-Type Ultra-Centrifuge.	108/II
Rostagni, A., et al.	Measuring Low Vapour Pressures.	79/II
Saharia, V. N., et al.	Construction of a New Combined Vacuum Furnace and Mass Spectrometer for Universal Use.	105/II
St. Clair, H. W., et al.	Low-Pressure Distillation of Zinc, from Al-Zn Alloy.	34/III
Sanders, J. V., et al.	Apparatus for the Study by Electron Diffraction of the Effect of Temperature on Surface Films.	63/II
Santiago, E. B., et al.	Industrial Applications of High Vacuum Techniques.	20/I
Saunders, L., et al.	Improvements in the Manufacture of Metallic Titanium.	76/III
Schaeffer, G. W., et al.	Surface Hardening of Ferrous Metal.	73/III
Scheer, J. W. A., van der	A Universal Power Supply Circuit for Use with Ionization Gauges.	60/II
Schlechten, A. W.	Vacuum Metallurgy—A New and Growing Industry.	71/III
Schlechten, A. W., et al.	Vacuum Treatment of Parkes' Process Crusts on a Pilot-Plant Scale.	120/III
Schlesinger, H., et al.	Surface Hardening of Ferrous Metals.	73/III
Schneider, M.	Metallising by High Vacuum.	1/III
Schofield, F.	A New Method of Preparing Paste and Powder Specimens for the Electron Microscope.	103/II
Schultz, K., et al.	Fractionation of Blood Plasma.	9/IV
Schulz, L. G.	An Interferometric Method for Accurate Thickness Measurements of Thin Evaporated Films.	92/III
Scott, G. D., et al.	The Thickness Measurement of Thin Films by Multiple-Beam Interferometry.	51/III
Scott, G. D., et al.	Continuous Observations with the Electron Microscope of the Formation of Evaporated Films of Silver, Gold and Tin.	98/III
Scott, N. W., et al.	A 72″ High Vacuum Evaporator and its Glow Discharge Equipment.	107/III
Sears, G. W., et al.	An Effective Vacuum Cut-Off.	75/II
Seegers, W. H., et al.	Changes in the Reactivity of Purified Prothrombin after Freeze-Drying.	33/IV
Sennett, R. S., et al.	The Thickness Measurement of Thin Films by Multiple-Beam Interferometry.	51/III
Sennett, R. S., et al.	Continuous Observations with the Electron Microscope of the Formation of Evaporated Films of Silver, Gold and Tin.	98/III
Shepherd, M., et al.	Mass Spectrometry.	16/II

Cumulative Author Index

Contd.

(For Abstract Nos. 1/I — 19/I; 1/II — 43/II; 1/III — 48/III; 1/IV — 22/IV see January Issue)
(For Abstract Nos. 20/I — 54/I; 44/II — 83/II; 49/III — 88/III; 23/IV — 40/IV see April Issue)

Author	Title of Publication	Abstr. No.
Sherwood, D. W., et al.	Leak Testing Device.	78/II
Shiflett, C. H., et al.	A System for the Collection and Purification of Radon by Remote Control.	83/II
Siegel, R.	Pumped Rectifiers in 600-volt Transportation Service.	51/II
Siggia, S., et al.	Direct Determination of Oxygen in Compounds Containing Carbon, Hydrogen and Oxygen.	51/I
Simon, I., et al.	Dry Metallic Friction as a Function of Temperature Between 4.2°K and 600°K.	68/I
Skeen, J. R.	Antibiotics.	49/I
Skinner, G. B., et al.	Vapour Pressure of Inorganic Substances V. Zirconium between 1,949 2,054°K.	74/I
Slater, L. E., et al.	New Evaporator Robotized for High Output Efficiency.	70/III
Sloane, J. F., et al.	New Techniques in Auto-Radiography.	10/IV
Slobod, R. L.	Low Temperature Separation of Ethane from Methane and Air.	43/IV
Smith, C. C., et al.	A Simplified Design for a Molecular Still of the Horizontal Type.	38/III
Smith, D. M., et al.	Guided Free-Film Distillation Apparatus.	82/III
Smith, L. P., et al.	Mass Spectrometric Study of Solids. I. Preliminary Study of Sublimation Characteristics of Oxide Cathode Materials.	35/IV
Smithson, G. R., et al.	Detection and Measurement of Organic Vapours.	2/IV
Snyder, F. L.	Insulation for High Voltage Apparatus.	7/I
Soc. Immobiliere & Financiere du Parc, et al.	Process of Preparing Coffee Extract.	132/III
Sowman, H. G., et al.	A Quenching Furnace for High Temperature Studies of Small Specimens.	117/III
Spence, L. U.	Falling-Film High Vacuum Still.	83/III
Spendlove, M. J., et al.	Low-Pressure Distillation of Zinc from Al-Zn Alloy.	34/III
Spiers, L., et al.	The Pirani Effect in a Thermionic Filament as a Means of Measuring Low Pressures.	4/II
Srivastava, B. N., et al.	Construction of a New Combined Vacuum Furnace and Mass Spectrometer for Universal Use.	105/II
Stanley, J. K., et al.	Determination of Oxygen in Zirconium Metal by the Vacuum Fusion Method.	123/III
Stanworth, J. E.	A Nickel-Chromium-Iron Alloy for Sealing to Glass.	52/IV
Steckelmacher, W., et al.	Improvements in Measuring Devices Dependent on Radiometer Action.	6/II
Steckelmacher, W., et al.	Automatic Regulation of Thermionic Emission.	17/IV
Stefaniak, J. J., et al.	Antibiotics Development.	25/III
Steidlitz, M. E., et al.	A System for the Collection and Purification of Radon by Remote Control.	83/III
Stephens, W. W., et al.	Production of Malleable Zirconium.	35/III
Stewart, D. W., et al.	Mass Spectrometric Analysis of Low Concentrations of Vapours in Air.	26/IV
Stoltenberg, C. R.	High Speed Mercury Diffusion Pump.	49/II
Sturr, J.	How to Metallize Glass and Other Ceramics.	64/III
Summers-Smith, D., et al.	A Laboratory Arc-Furnace for Melting Alloys Containing the Refractory Transition Metals.	78/III
Sylvania Electro Products Inc.	Metal Insert for Vacuum-Tight Sealing.	40/IV
Tatum, E. L., et al.	A Simplified Method of Lyophilizing Micro-organisms.	46/IV
Ternisien, J.	Electron Diffraction.	18/II
Thompson, A. R.	Vacuum Crystallizers.	82/II
Tomlinson, J. W., et al.	A Vacuum Furnace for Use in the Temperature Range of 1,000°-2,000°C.	32/III
Trevan, J. W., et al.	Preservation of Fungal Sporophores.	11/IV
Trillat, J. J., et al.	Checking the Purity of Hydrocarbon Liquids by Means of Electron Diffraction.	82/I
Trillat, J. J., et al.	An Apparatus for the Study of Insulating Surfaces by Means of Electron Diffraction.	62/II
U.S. Atomic Energy Commission, et al.	Leak Testing Device.	78/II
U.S. Atomic Energy Commission, et al.	Vacuum Pumping Method with Mechanical Oil Sealed Pumps.	89/II
U.S. Atomic Energy Commission, et al.	Ion Gauge.	95/II
U.S. Atomic Energy Commission, et al.	Surface Hardening of Ferrous Metals.	73/III
Vasicek, A.	Achromatizing of Thin Films.	71/I

Vol. 1 1951

Cumulative Author Index

Contd.

(For Abstract Nos. 1/I — 19/I; 1/II — 43/II; 1/III — 48/III; 1/IV — 22/IV see January Issue)
(For Abstract Nos. 20/I — 54/I; 44/II — 83/II; 49/III — 88/III; 23/IV — 40/IV see April Issue)

Author	Title of Publication	Abstr. No.
Veith, M. W.	On the Production of Signals in Television Camera Tubes. The Conductron Tube.	39/IV
Verleger, H., et al.	Low-Reflectance Coatings and the Sensitivity Curve of the Eye.	72/I
Viens, G. E., et al.	Refining Lithium by Vapourization at Low Pressure.	121/III
Wade, F.	The Measurement of High Vacuum by Electrical Methods.	90/II
Wagener, S.	A Method for Measuring the Efficiency of Getters at Low Pressures.	23/I
Walker, L. H., et al.	Experimental Falling Film-Evaporator.	112/III
Wallace & Tiernan Products Inc.	Improvements i o.r.t. Absolute Pressure Controlling Apparatus.	77/II
Ward, A. H.	Methods of Estimating Moisture.	84/I
Warner, F. E., et al.	Manufacture of Mononitrotoluene.	37/III
Warren, J.	Measurement of Appearance Potentials of Ions Produced by Electron Impact Using a Mass Spectrometer.	6/IV
Washburn, H. W., et al.	Method for Treating Filaments of Ionization Gauges.	58/II
Watson, J. H. L., et al.	Motion Picture Studies of Electron Bombardment of Colloidal Crystals.	65/II
Watt, P. R., et al.	The Concentration of Tocopherols from Natural Sources by Molecular Distillation.	81/III
Waugh, C. T.	Metal Backed Television Picture Tubes.	49/IV
Weeks, J. R.	Metallised Paper Capacitors.	63/I
Weinstein, A., et al.	A Geiger-Counter Mounting for Continuous Counting of Gas-Phase Radioactivity.	78/I
Wells, J. E.	The Accurate Determination of Carbon in Low Carbon Steels.	72/III
Wenzel, F. W., et al.	New Evaporator Robotized for High Output Efficiency.	70/III
Weston, G. F., et al.	The Spectral Emittance of Nickel- and Oxide-Coated Nickel Cathodes.	51/IV
Wheeler, A., et al.	Determination of Heats of Adsorption Using Metal Films.	59/III
White, A. B., et al.	Improvements i.o.r.t. the Heating of Rotating Workpieces by Electromagnetic Induction.	128/III
White, G. K., et al.	A Medium Capacity Helium Liquefier.	119/II
Whitehead, W. L. el al.	Vacuum Differential Thermal Analysis	87/III
Whiteley, R. S., et al.	Lubricants Produced by Reactions in Glow Discharge.	131/III
Wiblin, E. R., et al.	The Harwell Time-of-Flight Neutron Spectrometer.	107/II
Wiener, G., et al.	Determination of Oxygen in Zirconium Metal by the Vacuum Fusion Method.	123/III
Wilder, M. P., et al.	Getter Materials for Electron Tubes.	24/I
Wiley, H., et al.	Method for Treating Filaments of Ionization Gauges.	58/II
Wilkinson, P. G., et al.	Particle Size of Evaporated Gold.	15/III
Williams, R. C., et al.	The Use of Spraying Methods and of Volatile Suspending Media in the Preparation of Specimens for Electron Microscopy.	20/II
Wilson, B. A., et al.	Reflectivity of Thin Silver Films and their Use in Interferometry.	55/III
Wilt, W. A., et al.	Molecular Still.	126/III
Winkle, M. van., et al.	Vapour-Liquid Equilibria at Sub-atmospheric Pressures.	125/III
Witts, G. H., et al.	An Automatic Control of High Vacuum Systems.	39/II
Witty, R.	Characteristics of Diffusion Pumps.	47/II
Wood, W. D.	Shop Facilities for Testing Pressure Instruments.—Part I.	52/II
Zahm, G. G., et al.	Apparatus and Method for Evaporation and Concentration of Liquids.	69/III
Zemansky, M. W., et al.	Design and Performance of a Joule-Kelvin Helium Liquefier.	120/II

Abbreviations and Publishers of Journals Quoted in this Volume

An. Mec. Elect.	Anales de Mecanica y Electricidad, Reina 31, Madrid, Spain.
Analyst	The Analyst, The Society of Public Analysts and Other Analytical Chemists, 7-8, Idol Lane, London, E.C.3, England.
Analyt. Chem.	Analytical Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
ASEA Journal	ASEA Journal, Allmänna Svenska Elektriska Aktiebolaget, Vasteras, Sweden.
Aust. J. Appl. Phys.	Australian Journal of Applied Physics, Commonwealth Scientific and Industrial Research Organisation, 314, Albert Street, East Melbourne, C.2, Victoria, Australia.
Brit. J. Appl. Phys.	British Journal of Applied Physics, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
Canad. J. Res.	Canadian Journal of Research, National Research Council, Ottawa, Canada.
Canad. Min. Metall. Bull.	The Canadian Mining and Metallurgical Bulletin, Canadian Institute of Mining and Metallurgy, 906, Drummond Building, Montreal, Quebec, Canada.
Ceramic Industr.	Ceramic Industry, Industrial Publication Inc., 5, S. Wabash Avenue, Chicago 3, Ill., U.S.A.
Chem. Age	Chemical Age, Benn Bros., Bouverie House, 154, Fleet Street, London, E.C.4, England.
Chem. Engng.	Chemical Engineering, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Chem. Wk.	Chemical Week, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Chem. Ing. Tech.	Chemie Ingenieur Tecknik, Chemie Verlag, Weinheim/Bergstrasse, Hauptstrasse 129, Germany.
Chem. & Drugg. Export Rev.	Chemist and Druggist Export Review, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Chem. & Industr.	Chemistry and Industry, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
C.R. Acad. Sci., Paris	Comptes Rendus des Séances de l'Academie des Science, Messrs. Gauthier-Villar, Quai des Grands-Augustine 55, Paris, France.
Discuss. Faraday Soc.	Discussions of the Faraday Society, The Faraday Society, Aberdeen University Press, Ltd., 6, Upper Kirkgate, Aberdeen, Scotland.
Elect. Rev.	Electrical Review, Electrical Review Publications Ltd., Dorset House, Stamford Street, London, S.E.1, England.
Electronic Engng.	Electronic Engineering, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Electronics	Electronics, McGraw-Hill Publishing Co., Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Elektrotech. Z.	Elektrotechnische Zeitschrift, Verband Deutscher Elektrotechniker (VDE) Verlag, Wuppertal, Germany.
Engineer	The Engineer, Morgan Bros. (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Engng. Min. J.	Engineering and Mining Journal, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Food Industr.	Food Industries, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Food Manuf.	Food Manufacture, Leonard Hill, Ltd., 17, Stratford Place, London, W.1, England.
Foto-Kino-Technik	Foto-Kino-Technik, Foto-Kinotechnik G.m.b.h., Berlin-Borsigwalde, Eichborndamm 141-167, Germany.
Foundry	Foundry, Penton Publishing Co., Penton Bldg., Cleveland 13, Ohio, U.S.A.
Gen. Elect. Rev.	General Electrical Review, General Electric Co., 1, River Road, Schenectady 5, N.Y., U.S.A.

Vol. 1 1951

Abbreviations and Publishers of Journals Quoted in this Volume

Contd.

Il Nuovo Cimento	Il Nuovo Cimento, Nicola Zanichelli S. A, Via Irnerio 34, Bologna, Italy.
Industr. Chem.	Industrial Chemist, St. Margaret's Technical Press, Ltd., 33, Tothill Street, London, S.W.1, England.
Industr. Engng. Chem.	Industrial and Engineering Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Instrumentation	Instrumentation, Minneapolis-Honeywell Regulator Co. (Industrial Division), Wayne and Windrim Avenues, Philadelphia 44, Pa., U.S.A.
Instruments	Instruments, Instruments Publishing Co. Inc., 921, Ridge Avenue, Pittsburgh 12, Pa., U.S.A.
Int. Chem. Engng.	International Chemical Engineering and Process Industries, Leonard Hill, Ltd., 17, Stratford Place, London, W.1, England.
J. Amer. Ceramic Soc.	Journal of the American Ceramic Society, American Ceramic Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
J. Amer. Chem. Soc.	Journal of the American Chemical Society, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
J. Amer. Pharm. Ass.	Journal of the American Pharmaceutical Association, American Pharmaceutical Association, 2215, Constitution Avenue, N.W., Washington 7, D.C., U.S.A.
J. Appl. Phys.	Journal of Applied Physics, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Chem. Educ.	Journal of Chemical Education, Div. of Chemical Education of the American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
J. Inst. Fuel	Journal of the Institute of Fuel, Institute of Fuel, 18, Devonshire Street, Portland Place, London, W.1, England.
J. Iron & Steel Inst.	Journal of the Iron and Steel Institute, Iron and Steel Institute, 4, Grosvenor Gardens, London, S.W.1, England.
J. Metals	Journal of Metals, American Institute of Mining and Metallurgical Engineers, Inc., 29, West 39th Street, New York 18, N.Y., U.S.A.
J. Opt. Soc. Amer.	Journal of the Optical Society of America, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Phys. Radium	Journal de Physique et le Radium, Societie Francaise de Physique, Paris, France.
J. Phys. Soc., Japan	Journal of the Physical Society, Japan, Faculty of Science, University of Tokyo, Japan.
J. Rech. Centr. Nat. Rech. Sci.	Journal des Recherches du Centre National de la Recherche Scientifique, 13, Quai Anatole-France, Paris (VIIe), France.
J. Sci. Food Agric.	Journal of the Science of Food and Agriculture, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
J. Sci. Instrum.	Journal of Scientific Instruments, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
J. Soc. Chem. Industr.	Journal of the Society of Chemical Industry, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
Light Metals	Light Metals, Temple Press, Ltd., Bowling Green Lane, London, E.C.1, England.
Mach. Lloyd	Machinery Lloyd, Continental and Overseas Organisation, Ltd., 6, Cavendish Place, Regent Street, London, W.1, England.
Metal Prog.	Metal Progress, The American Society for Metals, 7301, Euclid Avenue, Cleveland 3, Ohio, U.S.A.
Metallurgia	Metallurgia, Kennedy Press, Ltd., 31, King Street West, Manchester 3, England.
Microtecnic	Microtecnic, Scriptar S.A., 23, Avenue de la Gare, Lausanne, Switzerland.

Abbreviations and Publishers of Journals Quoted in this Volume
Contd.

Mod. Plastics	Modern Plastics, Modern Plastics Inc., 122, E. 42nd Street, New York 17, N.Y., U.S.A.
Mycologia	Mycologia, Mycological Society of America, Prince and Lemon Streets, Lancaster Pa., U.S.A.
Nature (Lond.)	Nature, Macmillan and Co., St. Martin's Street, London, W.C.2, England.
Nucleonics	Nucleonics, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Optica Acta	Optica Acta, International Commission of Optics, 3, Boulevard Pasteur, Paris (15e), France.
Optik	Optik, Wissenschaftliche Verlagsgesellschaft m.b.H., Stuttgart, Germany.
Philips Techn. Rev.	Philips Technical Review, Philips Electrical Ltd., Century House, Shaftesbury Avenue, London, W.C.2, England.
Phys. Rev.	Physical Review, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
Proc. I.R.E.	Proceedings of the Institute of Radio Engineers, Institute of Radio Engineers, 1, East 79th Street, New York 21, N.Y., U.S.A.
Proc. Nat. Inst. Sci., India	Proceedings of the National Institute of Sciences, India, National Institute of Sciences of India, Delhi, India.
Proc. Phys. Soc.	Proceedings of the Physical Society, Physical Society, 1, Lowther Gardens, Prince Consort Road, London, S.W.7, England.
Proc. Soc. Exper. Biol., N. Y.	Proceedings of the Society for Experimental Biology and Medicine, College of the City of New York, New York, 31, N.Y., U.S.A.
Rech. Aéron.	La Recherche Aéronautique, Office Nationale d'Etudes et des Recherches Aéronautiques, 3, Rue Leon Bonnat, Paris (16e), France.
Research	Research, Butterworths Scientific Publications, Ltd., 4-6, Bell Yard, Temple Bar, London, W.C.2, England.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
Science	Science, American Association for the Advancement of Science, 1515, Massachusetts Avenue N.W., Washington 5, D.C., U.S.A.
Science Abstracts	Science Abstracts, Institution of Electrical Engineers, Savoy Place, London, W.C.2, England.
Sci. Rep. Res. Inst. Tohoku Univ.	Science Reports of the Research Institute of Tohoku University, Research Union Tohoku University, c/o Institute of Iron and Steel and other Metals, 75, Katahra Machi Sendai, Japan.
Slabopr. Obz.	Slaboproudy Obzor, Vocelova 3, Prague XII, Czechoslovakia.
Taylor Technol.	Taylor Technology, Taylor Instrument Co., Rochester (1), New York, U.S.A.
Telev. Engng.	Television Engineering, Bryan Davis Publishing Inc., 52, Vanderbilt Avenue, New York 17, N.Y., U.S.A.
The Times (Industr. Suppl.)	"The Times" Industrial Supplement, "The Times" Publishing Co., Ltd., Printing House Square, London, E.C.4, England.
The Times (Science Suppl.)	"The Times" Science Supplement, "The Times" Publishing Co., Ltd., Printing House Square, London, E.C.4, England.
Trans. Soc. Instrum. Technol.	Transactions of the Society of Instrument Technology, Society of Instrument Technology, 80, Bishopsgate, London, E.C.3, England.
Le Vide	Le Vide, Societe Francaise des Ingenieurs des Techniciens du Vide, 44, Rue de Rennes, Paris (VIe), France.
Westinghouse Engr.	Westinghouse Engineer, Westinghouse Electric Corporation, 306, Fourth Avenue, P.O. Box 1017, Pittsburgh 30, Pa., U.S.A.
Z. Angew. Phys.	Zeitschrift fur Angewandte Physik, c/o Springer Verlag/Berlin-Charlottenburg, Jebensstrasse 1, Germany.

Vol.
1
1951